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UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 C.F.R. § 1.53(b))

Attorney Docket No. 715-1-060 CIP

First Inventor or Application Identifier John Liskowitz

Title Method For Preparing Fly Ash For High.

Express Mail Label No. EL267940990US

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

1. ☒ * Fee Transmittal Form (e.g., PTO/SB/17)
(Submit an original and a duplicate for fee processing)
2. ☒ Specification [Total Pages 91]
(preferred arrangement set forth below)
 - Descriptive title of the invention
 - Cross References to Related Applications
 - Statement Regarding Fed sponsored R & D
 - Reference to Microfiche Appendix
 - Background of the invention
 - Brief Summary of the invention
 - Brief Description of the Drawings (if filed)
 - Detailed Description
 - Claim(s)
 - Abstract of the Disclosure
3. ☒ Drawing(s) (35 U.S.C. 113) [Total Sheets 23]
4. Oath or Declaration [Total Pages 3]
 - a. ☐ Newly executed (original or copy)
 - b. ☐ Copy from a prior application (37 C.F.R. § 1.63(d))
(for continuation/divisional with Box 16 completed)
 - i. ☐ DELETION OF INVENTOR(S)
Signed statement attached deleting inventor(s) named in the prior application, see 37 C.F.R. §§ 1.63(d)(2) and 1.33(b).

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 - a. ☐ Computer Readable Copy
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7. ☐ Assignment Papers (cover sheet & document(s))
8. ☐ 37 C.F.R. § 3.73(b) Statement ☐ Power of Attorney
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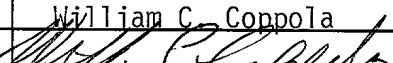
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Prior application information. Examiner M. Marchesani Group / Art Unit: 1755

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FIELD OF THE INVENTION

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BACKGROUND OF THE INVENTION

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1960's and 70's the cost of ash disposal was typically less than \$1.00 per ton. However, due to the more stringent environmental regulations starting in the late 1970's, the cost of ash disposal has rapidly increased to from \$2.00 to \$5.00 per ton and is still rising higher (Bahor and Golden, 1984, Proceedings, 2nd International Conference on Ash Technology and Marketing, London, pp. 133-136). The shortage of landfill due to environmental concerns has further escalated the disposal cost. The Environmental Protection Agency (EPA) estimated in 1987 that the total cost of waste disposal at coal fired power plants ranged from \$11.00 to \$20.00 per ton for fly ash and bottom ash (Courst, 1991, Proceedings: 9th International Ash Use Symposium, 1:21-1 to 21-10). This increasing trend of disposal cost has caused many concerns and researchers are urgently seeking means for better utilization of fly ash. One potential outlet for fly ash is incorporation in concrete or mortar mixtures.

Fly ash is used in concrete in two distinct ways, one as a replacement for cement and the other as a filler. The first use takes advantage of the pozzolanic properties of fly ash, which, when it reacts with lime or calcium hydroxide, can enhance the strength of cementitious composites. However, fly ash is relatively inert and the increase in compressive strength can take up to 90 days to materialize. Also, since fly ash is just a by-product from the power industry, the quality of fly ash has always been a major concern to the end users in the concrete industry.

Incorporation of fly ash in concrete improves workability and thereby reduces the water requirement with respect to the conventional concrete. This is most beneficial where concrete is pumped into place. Among numerous other beneficial effects are reduced bleeding, reduced segregation, reduced permeability, increased plasticity, lowered heat of hydration, and increased setting times (ACI Committee 226, 1987, *supra*). The slump is higher when fly ash is used (Ukita et al., 1989, SP-114, American Concrete Institute, Detroit, pp.219-240).

However, the use of fly ash in concrete has many drawbacks. For example, addition of fly ash to concrete results in a product with low air entrainment and low early strength development.

As noted above, a critical drawback of the use of fly ash in concrete is that initially the fly ash significantly reduces the compressive strength of the concrete. Tests conducted by Ravindrarajah and Tam (1989, Fly Ash, Silica Fume, Slag, and Natural Pozzolans in Concrete, SP-114, American Concrete Institute, Detroit, pp. 139-155) showed that the compressive strength of fly ash concrete at early ages are lower than those for the control concrete, which is a general property of concrete or mortar when fly ash is added. Most of the reported studies tend to show a lower concrete strength due to the presence of fly ash; none has yet suggested a solution to actually enhance the property of concrete economically. Yet, for fly ash to be used as a replacement for cement, it must be comparable to cement in terms of strength contribution at a point useful in construction. As a practical matter, this means that the fly ash concrete must reach an acceptable compressive strength within about 2 weeks.

Swamy (1984, Proceedings, 2nd International Conference on Ash Technology and Marketing, London, pp. 359-367) showed that 30% replacement by weight, and inclusion of a high dose of a superplasticizer, yielded concrete with material properties and structural behavior almost identical to those of concrete of similar strength without fly ash. However, due to the high cost of superplasticizer, mix proportions were not economical.

Fly ashes from different sources may have different effect to concrete. The same fly ash may behave differently with Portland cements of different types (Popovics, 1982, ACI J. Proceedings 79:43-49), since different types of Portland cement (type I to V) have different chemical composition. Other factors relating to the effects of fly ash on concrete that are not presently understood are lime availability, the rate of solubility and reactivity of the glassy phase in different fly ash, and the proper mix proportion to ensure early strength development of fly ash concrete.

Fly ash particles are typically spherical, ranging in diameter from 1 to 150 microns (Berry and Malhotra, 1980, ACI J. Proceedings 77:59-73). Aitcin et al. (1986, Fly Ash, Silica Fume, Slag, and Natural Pozzolans in Concrete, SP-91, American Concrete Institute, Detroit, pp. 91-113) showed that if the average diameters, D_{50} , of fly ash are smaller, the surface area of the fly ash will be larger than those with larger average diameters.

Many factors affect the size or average diameter of fly ash, including storage conditions, ash collection processes, and combustion conditions. Combustion conditions are perhaps most important, because these determine whether carbon remains in the ash or if combustion is complete.

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There are two main forms of combustion: dry bottom boiler combustion and wet bottom boiler combustion. The main difference between the two types of boiler is that wet bottom boilers reach the fusion temperature of ash, thus resulting in fly ash with greater glass characteristics.

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There are generally two methods known to measure the fineness of fly ash. The first is by measuring the residue on the 45 micron (No. 325 sieve), which is the method used in the United States. The second method is the surface area method by air permeability test.

Lane and Best (1982, *Concrete International: Design & Construction* 4:81-92) suggested

15 that 45 microns sieve residue is a consistent indicator of pozzolanic activity. For use in concrete or mortar, ASTM C 618 (1990, ASTM C 618-89a, *Annual Book of ASTM Standards*, Vol. 04.02) specifies that not more than 34% by weight of a given fly ash be retained on a 45 microns sieve. However, Ravina (1980, *Cement and Concrete Research* 10:573-580) reported that specific surface area provides a more accurate indicator of
20 pozzolanic activity.

Research carried out by Ukita et al. (1989, *supra*) purported that as the percentage of finer particles, *i.e.*, those particles ranging from diameters of 1 to 20 microns, in concrete increases, the corresponding strength gain is notable. Similar observations have been
25 reported by Giergiczny and Werynska (1989, *Fly Ash, Silica Fume, Slag, and Natural Pozzolans in Concrete*, SSP-114, American Concrete Institute, Detroit, pp. 97-115).

Both of the groups mentioned above describe results with fly ash of disparate characteristics and sources, but did not include controls for these variable. Thus, although
30 the emphasis of these reports is on the performance of finer particle fly ashes, the variables introduced into the studies lead to reservations with respect to any conclusions that may be drawn. In particular, Ukita et al. (1989, *supra*) collected fly ash from different locations. However, an earlier report demonstrated that fly ashes collected from different locations

have different chemical properties (Liskowitz et al., 1983, "Sorbate Characteristic of Fly Ash," Final Report, U.S. Dept. of Energy, Morgantown Energy Technology Center, p. 211). Giergiczny and Werynska (1989, *supra*) ground the original fly ash into different sizes. Grinding can add metal particles into the fly ash, and also tends to yield unnaturally shaped particles of fly ash. Thus, these reports fail to provide conclusive information about the effect of fine particle size on the properties imparted by fly ash.

Berry et al. (1989, Fly Ash, Silica Fume, Slag, and Natural Pozzolans in Concrete, SP-114, American concrete Institute, Detroit, pp. 241-273) studied the properties of fly ash with particle size smaller than 45 microns, so called "beneficiated" fly ash, in mortar. Fly ashes of this particle size showed improved pozzolanic activity, reduced water demand and enhanced ability to reduce alkali-aggregate reactivity.

Although beneficiated fly ash seem to show promising results in terms of improved performance of mortar, other researchers concluded otherwise when used in concrete. Giaccio and Malhotra (1988, Cement, Concrete, and Aggregates 10:88-95) also conducted the test using the beneficiated fly ashes. They showed that the concrete made with ASTM type I cement, the use of beneficiated fly ash and condensed silica fume did little to enhance the properties of concrete compared with the raw fly ash.

As explained above, efforts have been made to grind fly ash recovered from boilers so that they have a particle size in order to increase their pozzolanic activity and thus substitute for pozzolanic materials such as cement having comparable contribution at a point useful in construction as concrete that does not use fly ash. Grinding the fly ash has met with only limited success. There are numerous means to grind fly ash so that it can be used as a pozzolanic material. One such method involves grinding the fly ash with a grinding medium, such as zirconium silicate particles. In particular, Perry's Chemical Engineers Handbook, 6TH Edition, teaches that in order to grind fly ash into particles having an appropriate size for use as a substitute for cement in concrete or mortar, the volume of fly ground should be equal to or greater than the void volume of the grinding material [page 8-32 of Perry's Handbook, 6th Edition]. However, these grinding methods have met with only limited success in that the yield of fly ash particles having a diameter less than about 12 μm is low. As a result, much fly ash is wasted, and the grinding process is not

economical.

Accordingly, what is needed are methods of grinding fly ash into particles that have a size that permits its use in concrete or mortar that undergo strength development comparable to,
5 or superior to concrete and mortar comprising no fly ash, and which are readily used in the construction industry.

There is also a need utilize all of the fly ash produced in coal-dust fired boilers, thus decreasing the amount of waste produced by boilers, and decreasing the amount of cement
10 used in the construction industry.

There is a further need to process fly ash efficiently to provide about 100% yield of useful product in desirable production quantities.

15 There is a further need in the art for high strength concrete and mortar containing fly ash.

There is yet a further need in the art for the utilization of fly ash generated during coal combustion.

20 These and other needs in the art are addressed in the instant application.

The citation or identification of any reference in this application shall not be construed as an admission that such reference is available as prior art to the present invention.

25 **SUMMARY OF THE INVENTION**

The present invention relates to hardenable mixtures comprising processed fly ash of a defined fineness as a replacement for cement in cementitious materials, which hardenable mixtures achieve compressive strength that is about equal to or greater than the
30 compressive strength of the same hardenable mixture without fly ash in a time period that is acceptable for construction and other applications. In particular embodiments, the hardenable mixture can be concrete or mortar, as hereinafter defined.

The invention is related to the invention disclosed and claimed in U.S. Patent No. 5,624,491 and in International Patent Publication No. WO 95/32423, published November 30, 1995, of PCT International Patent Application No. PCT/US95/06182, both entitled "IMPROVED COMPRESSIVE STRENGTH OF CONCRETE AND MORTAR

- 5 CONTAINING FLY ASH," each of which is incorporated herein by reference in their entireties. However, the present invention is based on the discovery that fly ash having desirable characteristics, such as a fineness modulus as defined in U.S. Patent 5,624,491 and International Publication No. WO 95/32423 (PCT/US95/06182), can be prepared by dry processing fly ash from boilers so as to shift the entire distribution of sizes to a desired
- 10 range, while retaining substantially uniform spherical shape of the processed fly ash. This invention advantageously avoids the need for size fractionation, *e.g.*, by air classification, and provides a product with approximately 100% yield in desirable production quantities that, when incorporated in a hardenable mixture, such as concrete or mortar, demonstrates compressive strength properties that are equal to or better than fractionated fly ash achieves
- 15 with such classification methodology, such as air classification.

Broadly, the present invention extends to fly ash characterized by:

- a. substantially uniform spherical shape;
 - b. greater than about 90% of the particles have a diameter of less than 11 μm , greater
 - 20 than about 60% of the particles have a diameter of less than 5.5 μm , and greater than about 15% of the particles have a diameter of less than 1.375 μm ;
 - c. a median particle diameter of less than about 4.0 μm ; and
 - b. a range of particle diameters of from about 0.1 μm to about 70 μm .
- 25 Furthermore, the present invention extends to fly ash as described above, wherein greater than about 93% of the particles have a diameter of less than 11 μm , greater than about 70% of the particles have a diameter of less than 5.5 μm , and greater than about 18% of the particles have a diameter of less than 1.375 μm .
- 30 In a particular embodiment, the present invention extends to fly ash characterized by having a median particle diameter of less than about 3.0 μm .

Furthermore, the present invention extends to fly ash as described above, wherein the range

of particle diameters is from about 0.9 μm to about 62 μm .

Furthermore, any type of fly ash described herein has applications in the invention. In a particular embodiment, the fly ash is prepared by grinding unfractionated fly ash. Methods of grinding fly ash are set forth *infra*.

- 5 The present invention further extends to concrete and mortar which utilize fly ash. In particular, the present invention extends to a concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water. The cementitious materials of concrete and mortar of the invention comprise from about
 - 10 10% to about 50% by weight the fly ash characterized by:
 - a. substantially uniform spherical shape;
 - b. greater than about 90% of the particles have a diameter of less than 11 μm , greater than about 60% of the particles have a diameter of less than 5.5 μm , and greater than about 15% of the particles have a diameter of less than 1.375 μm ;
 - 15 c. a median particle diameter of less than about 4.0 μm ; and
 - b. a range of particle diameters of from about 0.1 μm to about 70 μm .

The concrete or mortar of this embodiment also comprises about 50% to about 90% by weight cement.
- 20 In addition, the present invention extends to a concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight fly ash characterized by:
 - 25 a. substantially uniform spherical shape;
 - b. greater than about 93 % of the particles have a diameter less than 11 μm , greater than about 70 % of the particles have a diameter less than 5.5 μm , and greater than about 18% of the particles have a diameter of less than 1.375 μm ;
 - 30 c. the median particle diameter is less than about 3.0 μm ; and

d. the range of particle diameters is from about 0.9 μm to about 62 μm , and about 50% to about 90% by weight cement.

Optionally, concrete of the invention as described above can further comprise silica fume,
5 glass fibers, or a combination thereof.

In another embodiment the present invention extends to a mortar comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise
10 from about 10% to about 50% by weight the fly ash and about 50% to about 90% by weight cement, wherein the fly ash is characterized by:

- a. substantially uniform spherical shape;
- b. greater than about 90% of the particles have a diameter of less than 11 μm , greater than about 60 % of the particles have a diameter of less than 5.5 μm , and greater
15 than about 15 % of the particles have a diameter of less than 1.375 μm ;
- c. a median particle diameter of less than about 4.0 μm ; and
- d. a range of particle diameters of from about 0.1 μm to about 70 μm .

In another embodiment, the present invention extends to a mortar comprising about 1 part
20 by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the fly ash and about 50% to about 90% by weight cement, wherein the fly ash is characterized by:

- a. substantially uniform spherical shape;
- 25 b. greater than about 93 % of the particles have a diameter less than 11 μm , greater than about 70 % of the particles have a diameter less than 5.5 μm , and greater than about 18% of the particles have a diameter of less than 1.375 μm ;
- c. the median particle diameter is less than about 3.0 μm ; and
- d. the range of particle diameters is from about 0.9 μm to about 62 μm , and
30 about 50% to about 90% by weight cement.

Optionally, a mortar of the invention can further comprise silica fume, glass fibers, or a combination thereof.

The present invention also extends to methods for preparing fly ash, so as to shift the size distribution of fly ash particles, such that the finished fly ash has the following characteristics:

- a. substantially uniform spherical shape;
- 5 b. greater than about 90% of the particles have a diameter of less than 11 μm , greater than about 60% of the particles have a diameter of less than 5.5 μm , and greater than about 15% of the particles have a diameter of less than 1.375 μm ;
- c. a median particle diameter of less than about 4.0 μm ; and
- d. a range of particle diameters of from about 0.1 μm to about 70 μm .

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The present invention encompasses numerous methods of producing fly ash having characteristics as described above. One such method involves a fluidized bed grinding process. In particular, the present invention extends to a method for preparing fly ash as described above, wherein the processing comprises grinding with a fluidized bed grinding

15 process such that the volume of fly ash ground in the process is less than the void volume of the grinding medium. As a result, the collisions between the fly ash and grinding medium occur at a sufficient frequency to grind the fly ash and shift the size distribution of fly ash particles. In a particular embodiment, the ratio of fly ash to grinding medium comprises about one part unfractionated fly ash to about seven parts grinding media (by

20 weight). In another embodiment, the ratio of fly ash to grinding medium comprises about one part fly ash to about 4 parts grinding medium, by volume. It has been discovered that fly ash serves as a lubricant during the grinding process. As a result, the high temperatures that would be expected in a situation where the volume of fly ash is less than the void volume of the grinding medium are not realized.

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Furthermore, numerous grinding medium have applications in the fluidized bed grinding process of the invention. Examples of grinding medium having applications herein include, but certainly are not limited to, zirconium silicate, carbon steel, chromium steel, or stainless steel, to name only a few.

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Furthermore, the present invention extends to concrete and mortar compositions. More specifically, in one embodiment, the present invention extends to a concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine

aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight fly ash and about 50% to about 90% by weight cement, wherein the fly ash is characterized by:

- 5 a) substantially uniform spherical shape;
- b) greater than about 90% of the particles have a diameter of less than 11 μm , greater than about 60% of the particles have a diameter of less than about 5.5 μm , greater than about 60 % of the particles have a diameter of less than 5.5 μm , and greater than about 15 % of the particles have a diameter of less than 1.375 μm ;
- 10 c) a median particle diameter of less than about 4.0 μm ; and
- d) a range of particle diameters of from about 0.1 μm to about 70 μm .

Furthermore, a concrete of the invention, as described above, can further comprise glass fibers, silica fume, or a combination thereof.

15

In addition, the present invention extends to concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight fly ash and about 50% to about 90% by weight cement, wherein the fly ash is characterized by:

20

- a. substantially uniform spherical shape;
- b. greater than about 93 % of the particles have a diameter less than 11 μm , greater than about 70 % of the particles have a diameter less than 5.5 μm , and greater than about 18% of the particles have a diameter of less than 1.375 μm ;
- 25 c. the median particle diameter is less than about 3.0 μm ; and
- d. the range of particle diameters is from about 0.9 μm to about 62 μm , and about 50% to about 90% by weight cement.

30

Naturally, a concrete of the invention as described above, can further comprise silica fume glass fibers, or a combination thereof.

Also, the present invention extends to a mortar comprising about 1 part by weight

cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight fly ash and about 50% to about 90% by weight cement, wherein the fly ash is characterized by:

- 5 a) substantially uniform spherical shape;
- b) greater than about 90% of the particles have a diameter of less than 11 μm , greater than about 60% of the particles have a diameter of less than about 5.5 μm , greater than about 60 % of the particles have a diameter of less than 5.5 μm , and greater than about 15 % of the particles have a diameter of less than 1.375 μm ;
- 10 c) a median particle diameter of less than about 4.0 μm ; and
- d) a range of particle diameters of from about 0.1 μm to about 70 μm .

In another embodiment, mortar of the invention comprises about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight fly ash and about 50% to about 90% by weight cement, wherein the fly ash is characterized by:

- a. substantially uniform spherical shape;
- b. greater than about 93 % of the particles have a diameter less than 11 μm , greater than about 70 % of the particles have a diameter less than 5.5 μm , and greater than about 18% of the particles have a diameter of less than 1.375 μm ;
- 20 c. the median particle diameter is less than about 3.0 μm ; and
- d. the range of particle diameters is from about 0.9 μm to about 62 μm , and about 50% to about 90% by weight cement.

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What's more, a mortar of the invention, as described above, can further comprise glass fibers, silica fume, or a combination thereof.

- 30 Moreover, the present invention extends to a method for preparing fly ash comprising processing fly ash so as to shift the size distribution to have the following characteristics:
 - a) substantially uniform spherical shape;
 - b) greater than about 90% of the particles have a diameter of less than 11 μm , greater

than about 60% of the particles have a diameter of less than about 5.5 μm , greater than about 60 % of the particles have a diameter of less than 5.5 μm , and greater than about 15 % of the particles have a diameter of less than 1.375 μm ;

- c) a median particle diameter of less than about 4.0 μm ; and
- 5 d) a range of particle diameters of from about 0.1 μm to about 70 μm .

In particular, the present invention extends to a method of preparing fly ash as described above, wherein the processing comprises grinding fly ash with a fluidized bed grinding process using a ratio of one part unfractionated fly ash with seven parts grinding medium
10 (by weight).

Numerous grinding media are readily available to the skilled artisan and have applications in a method of the invention. Particular examples include zirconium silicate, stainless steel, or carbon steel, to name only a few.

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Furthermore, fly ash ground in a method of the invention can be dry bottom boiler or wet bottom boiler fly ash.

Furthermore, the present invention extends to fly ash prepared so as to shift the size
20 distribution to have the following characteristics:

- a. substantially uniform spherical shape;
- b. greater than about 90% of the particles have a diameter of less than 12 μm , greater than about 50% of the particles have a diameter of less than 5 μm , and greater than about 15% of the particles have a diameter of less than 2.3 μm ;
- 25 c. a median particle diameter of less than about 6.0 μm ; and
- d. a range of particle diameters of from about 0.78 μm to about 30 μm .

In a particular embodiment, fly ash prepared to shift the size distribution as described above is by grinding the fly ash with a grinding medium in a non-expanded bed, wherein
30 the volume of fly ash is less than the void volume of the grinding medium. For example, in one embodiment of the invention, the ratio of fly ash to grinding medium in the non-expanded bed grinding process is about 1 part fly ash to about 4 parts grinding medium, by volume. In another embodiment, the ratio of fly ash to grinding medium comprises about 1

part fly ash to about 18 parts grinding medium, by weight. Naturally, numerous grinding medium are available to with the requisite specific gravity for using in producing fly ash as described above. Examples include, but certainly are not limited to carbon steel or stainless steel. In a particular embodiment of the invention, the grinding medium

5 comprises carbon steel spheres having a diameter of about 1/8 inch.

In yet another embodiment, the present invention extends to a concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight fly ash and about 50% to about 90% by weight cement, wherein the fly ash is characterized by:

- a) substantially uniform spherical shape;
 - b) greater than about 90% of the particles have a diameter of less than 11 μm , greater than about 60% of the particles have a diameter of less than about 5.5 μm , greater than about 60% of the particles have a diameter of less than 5.5 μm , and greater than about 15% of the particles have a diameter of less than 1.375 μm ;
 - c) a median particle diameter of less than about 4.0 μm ; and
 - d) a range of particle diameters of from about 0.1 μm to about 70 μm ,
- 20 wherein the fly ash is prepared by grinding fly ash with a grinding medium with a fluidized bed grinding process such that the volume of fly ash ground in the process is less than the void volume of the grinding medium. In a particular embodiment, the ratio of fly ash to grinding medium comprises about one part unfractionated fly ash to about seven parts grinding media (by weight).

25 In addition, the present invention extends to a concrete as described above, wherein the grinding medium comprises zirconium silicate, stainless steel or carbon steel. In addition, optionally, the concrete can further comprises glass fibers, silica fume, or a combination thereof. In a preferred embodiment, the grinding medium comprises zirconium silicate with a diameter of about 2-2.5 mm.

30 Moreover, the present invention extends to a mortar comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to

about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight fly ash and about 50% to about 90% by weight cement, wherein the fly ash is characterized by:

- a) substantially uniform spherical shape;
 - 5 b) greater than about 90% of the particles have a diameter of less than 11 μm , greater than about 60% of the particles have a diameter of less than about 5.5 μm , greater than about 60 % of the particles have a diameter of less than 5.5 μm , and greater than about 15 % of the particles have a diameter of less than 1.375 μm ;
 - c) a median particle diameter of less than about 4.0 μm ; and
 - 10 d) a range of particle diameters of from about 0.1 μm to about 70 μm ,
- wherein the fly ash is prepared by grinding fly ash with a grinding medium with a fluidized bed grinding process such that the volume of fly ash ground in the process is less than the void volume of the grinding medium. In a particular embodiment, the ratio of fly ash to grinding medium comprises about one part unfractionated fly ash to about seven parts
- 15 grinding media (by weight). Furthermore, just as with concrete of the invention, numerous grinding media can be used in a mortar of the invention. Examples of grinding medium having applications herein include, but are not limited to zirconium silicate, carbon steel, or stainless steel. In a particular embodiment, the grinding medium comprises zirconium silicate having a diameter of about 2-2.5 mm.

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Furthermore, the present invention extends to a mortar as described above, further comprising glass fibers, silica fume, or a combination thereof.

- In another embodiment, the present invention extends to a concrete comprising fly ash
- 25 prepared using a non-expanded grinding bed process. In particular, the present invention extends to a concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight fly ash and about 50% to
 - 30 about 90% by weight cement, wherein the fly ash is characterized by:
 - a. substantially uniform spherical shape;
 - b. greater than about 90% of the particles have a diameter of less than 12 μm , greater than about 50% of the particles have a diameter of less than 5 μm , and greater than

- about 15% of the particles have a diameter of less than $2.3\ \mu\text{m}$;
- c. a median particle diameter of less than about $6.0\ \mu\text{m}$; and
- d. a range of particle diameters of from about $0.78\ \mu\text{m}$ to about $30\ \mu\text{m}$.

- 5 The fly ash of this concrete of the invention is prepared by grinding the fly ash with a grinding medium in a non-expanded bed, wherein the volume of fly ash in the non-expanded bed is less than the void volume of the grinding medium. Numerous grinding medium have applications in a non-expanded bed grinding process involved in the concrete of the invention. In an embodiment, the ratio of fly ash to grinding medium comprises
- 10 about 1 part fly ash to about 4 parts grinding medium by volume. In another embodiment, the ratio of fly ash to grinding medium comprises about 1 part fly ash to about 18 parts grinding medium by weight. Particular examples of grinding medium include, but are not limited to carbon steel or stainless steel. In a particular embodiment of concrete, the grinding medium comprises carbon steel having a diameter of about $1/8$ inch.

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Moreover, the present invention extends to a concrete as described above, further comprises glass fibers, silica fume, or a combination thereof.

- Naturally, the present invention extends to a mortar comprising fly ash prepared using a
- 20 non-expanded grinding bed process. More specifically, the present invention extends to a mortar comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight fly ash and about 50% to about 90% by weight cement. The cementitious materials of mortar of the
- 25 invention comprise from about 10% to about 50% by weight fly ash and about 50% to about 90% by weight cement, wherein the fly ash is characterized by:

- a. substantially uniform spherical shape;
- b. greater than about 90% of the particles have a diameter of less than $12\ \mu\text{m}$, greater than about 50% of the particles have a diameter of less than $5\ \mu\text{m}$, and greater than
- 30 about 15% of the particles have a diameter of less than $2.3\ \mu\text{m}$;
- c. a median particle diameter of less than about $6\ \mu\text{m}$; and
- d. a range of particle diameters of from about $0.78\ \mu\text{m}$ to about $30\ \mu\text{m}$.

The fly ash of mortar of the invention is prepared by grinding the fly ash with a grinding

medium in a non-expanded bed, wherein the volume of fly ash in the non-expanded bed is less than the void volume of the grinding medium. In an embodiment of the invention, the ratio of fly ash to grinding medium in the non-expanded bed comprises about 1 part fly ash to about 4 parts grinding medium by volume. In another embodiment, the ratio of fly ash to grinding medium comprises about 1 part fly ash to about 18 parts grinding medium, by weight.

Numerous grinding media have applications in a mortar of the invention as described above. Examples include, but certainly are not limited to carbon steel or stainless steel.

10 In a particular embodiment, the grinding medium comprises carbon steel, having a diameter of about 1/8 inch.

Moreover, numerous types of fly ash have applications in methods, concrete, or mortar of the invention. Particular examples include dry bottom fly ash, or wet bottom fly ash.

15 An important advantage of the present invention relates to the improved compressive strength properties contributed by the processed fly ash hardenable mixtures. For example, in mortar prepared using processed fly ash of the invention to replace 25% of the cement, "cross-over" in compressive strength of the mortar compared with a control mortar occurs

20 within 7 to 14 days, and the compressive strength of the mortar containing processed fly ash is 50% greater than the control after 56 days.

As noted above, another advantage of the invention is that the process does not require fractionation, and allows for utilization of about 100% of the fly ash obtained from a boiler

25 operation to produce desirable production quantities of fly ash. Quantitative utilization of the fly ash avoids the need to dispose of undesirable fly ash fractions, which addressed the concerns of utilities for abatement of this otherwise undesirable pollutant.

A further advantage of the invention is that the grinding process appears to release

30 ammonia captured in the surface of fly ash produced from urea-treated coal (which is used to reduce NO_x emissions). The presence of ammonia in fly ash renders it unsuitable for use in concrete or mortar.

Yet another advantage of the present invention is that processing minimizes the effects of boiler conditions on the fly ash properties associated with boiler conditions and the degree of coal pulverization, such that the fly ash is desirable as a cement replacement in hardenable mixtures. Accordingly, fly ash can be dry bottom boiler fly ash or wet bottom boiler fly ash.

As is readily apparent to one of ordinary skill in the art, selection of starting material and grinding conditions are necessary to yield the fly ash of the invention. In a specific embodiment exemplified herein, unfractionated (unclassified) fly ash unexpectedly yields a superior processed fly ash product after grinding; a coarse fraction of fly ash does not yield the same quality processed fly ash product under the same grinding conditions.

Furthermore, one of ordinary skill in the art can readily use a method of the invention set forth below, particularly a method of grinding fly ash in a non-expanded bed, to produce fly ash readily usable in various industries, in desired production quantities, i.e., approximately three tons per hour.

In a further aspect, in a hardenable mixture, *i.e.*, concrete or mortar, of the invention, the fine aggregate may comprise sand and fly ash, wherein a ratio by weight of sand to fly ash is from about 4:1 to about 1:1. Preferably, the fly ash has a fineness modulus of less than about 600, wherein the fineness modulus is calculated as the sum of the percent of fly ash retained on sieves of 0, 1, 1.5, 2, 3, 5, 10, 20, 45, 75, 150, and 300 microns (as described in U.S. Patent 5,624,491 and International Publication No. WO 95/32423). In another embodiment, the fly ash used as fine aggregate is processed fly ash of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

FIGURE 1. Compressive strength of fly ash mortar prepared with the M8F95 air classified product, used to replace 25% of cement. Three series of mortars were prepared (square, lozenge, and triangle).

FIGURE 2. Compressive strength of fly ash mortar prepared with the M9F95 air classified product, used to replace 25% of cement. Three series of mortars were prepared

(square, lozenge, and triangle).

FIGURE 3. Compressive strength of fly ash mortar prepared with the M8F95 air
5 classified product, used to replace 35% of cement. Three series of mortars were prepared
(square, lozenge, and triangle).

FIGURE 4. Compressive strength of fly ash mortar prepared with the M9F95 air
classified product, used to replace 35% of cement. Three series of mortars were prepared
10 (square, lozenge, and triangle).

FIGURE 5. Compressive strength of fly ash mortar prepared with the M13N94 air
classified product, used to replace 25% of cement. Three series of mortars were prepared
(square, lozenge, and triangle).

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FIGURE 6 (prior art). Schematic diagram of the dry grinding process employed to
process the coarse fraction of air classified fly ash, and unfractionated fly ash. The Attritor
dry grinding process is achieved in a stationary tank by means of a rotating shaft and arms
that agitate the media into a random state of motion of internal porosity called kinematic
porosity. In this expanded condition, the media and particles are free to move, collide, and
20 impinge upon each other. (A) Kinematic porosity in expanded media. The revolving arm
hits grinding media. The media moves forward rapidly. The media collides with the
substrate, breaking up or reducing in size the product particle. (B) Media at rest when the
shaft is stationary. (C) Expanded media when the shaft is rotating.

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FIGURE 7. Analysis of the particle size distribution and mode for the coarse air classified
fly ash fraction (M8C95) prior to grinding.

FIGURE 8. Analysis of the particle size distribution and mode for the unfractionated fly
30 ash (M0C95) prior to grinding.

FIGURE 9. Compressive strength of fly ash mortar prepared with the M0G95 (ground,
unfractionated fly ash) product, used to replace 25% of cement. Three series of mortars

were prepared (square, lozenge, and triangle).

FIGURE 10. Compressive strength of fly ash mortar prepared with the M8CG95 air classified product after grinding, used to replace 25% of cement. Three series of mortars were prepared (square, lozenge, and triangle).

FIGURE 11. Compressive strength of fly ash mortar prepared with the M0G95 product, after grinding, used to replace 35% of cement. Three series of mortars were prepared (square, lozenge, and triangle).

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FIGURE 12. Compressive strength of control fly ash mortar prepared with cement without fly ash. Three series of mortars were prepared (square, lozenge, and triangle).

FIGURE 13. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in an expanded bed. 1050 lbs/hour of fly ash were ground with a grinding medium comprising 5.7 gallons (229 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting of grounder was 600.

FIGURE 14. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in an expanded bed. 1440lbs/hour lbs of fly ash were ground with a grinding medium comprising 5.7 gallons (229 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting was 600.

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FIGURE 15. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in an expanded bed. 1800lbs/hour of fly ash were ground with a grinding medium comprising 5.7 gallons (229 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting of the grinder was 600.

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FIGURE 16. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in a non-expanded bed. 1050 lbs/hour of fly ash were

5 **FIGURE 17.** Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in a non-expanded bed. 1440 lbs/hour of fly ash were ground with a grinding medium comprising 11.5 gallons (462 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting of the grinder was 400.

FIGURE 18. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in a non-expanded bed. 1800 lbs/hour of fly ash were ground with a grinding medium comprising 11.5 gallons (462 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting of the grinder was 400.

FIGURE 19. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in a non-expanded bed. 2000 lbs/hour of fly ash were ground with a grinding medium comprising 11.5 gallons (462 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting of the grinder was 400.

FIGURE 20. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in a non-expanded bed. 2200 lbs/hour of fly ash were ground with a grinding medium comprising 11.5 gallons (462 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting of the grinder was 400.

FIGURE 21. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in a non-expanded bed. 2280 lbs/hour of fly ash were ground with a grinding medium comprising 11.5 gallons (462 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting of the grinder was 400.

FIGURE 22. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in a non-expanded bed. 2700 lbs/hour of fly ash were ground with a grinding medium comprising 11.5 gallons (462 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting of the grinder was 400.

FIGURE 23. Histogram of particle size verses percentage of the volume of fly ash particles having a particular size ground in a non-expanded bed. 2700 lbs/hour of fly ash were ground with a grinding medium comprising 11.5 gallons (462 lbs) of carbon steel spheres having a diameter of 1/8 inch. The power setting for the grinder was 66 amps, and the RPM setting of the grinder was 400.

DETAILED DESCRIPTION OF THE INVENTION

As described above, the present invention relates to hardenable mixtures comprising processed fly ash of a defined fineness as a replacement for cement in cementitious materials, which hardenable mixtures achieve compressive strength that is about equal to or greater than the compressive strength of the same hardenable mixture without fly ash. The invention further provides for replacement of a portion of the fine aggregates in a hardenable mixture with fly ash of a defined fineness. In particular embodiments, the hardenable mixture can be concrete or mortar, as hereinafter defined.

Throughout this specification, where specific ratios, percentages, or proportions are mentioned, they are determined by weight and not by volume unless stated otherwise.

The present invention is based, in part, on the observation that processing fly ash from a boiler to shift the entire distribution range of fly ash particles to a smaller size, and to narrow that range, allows for essentially quantitative utilization of fly ash, and yields hardenable mixtures, *e.g.*, concrete and mortar, with a greater rate of compressive strength gain than other such compositions comprising fly ash, and crossover for greater compressive strength than such mixtures lacking fly ash after seven to fourteen days. These properties are independent of the source and chemical composition of fly ash, as the pozzolanic properties of the fly ash primarily depend on the degree of fineness of the fly

ash. It has been surprisingly found that processing fly ash by grinding to a defined fineness modulus as herein defined provides a high degree of quality control, regardless of the classification or combustion conditions of the fly ash.

- 5 Furthermore, the present invention is based upon the discovery that surprisingly, grinding a volume of fly ash with a grinding medium, wherein the fly ash has a volume less than the void volume of the grinding medium, permits increased collisions of fly ash particles with grinding medium. As a result, the fly ash is ground to a size which permits the use of fly ash in concrete and mortar of the invention, which have acceptable compressive strength
10 within about 2 weeks of the pouring of the concrete or mortar.

- In addition, the present invention is based upon the discovery that, unexpectedly, grinding fly ash in a non-expanded bed results in desirable production quantities of fly ash that is substantially spherical and shape, and greater than 90% of the fly ash particles have a size
15 less than 12 μm .

- As used herein, the phrase a "non-expanded bed" refers to a grinding bed which does not significantly increase in volume during the grinding process. More specifically, a grinder used to produce fly ash of the present invention, such as the HSA-30 attritor, comprises a
20 tank and a rotating shaft in the center of the tank, having arms protruding from the shaft. If these arms have a sufficient rotation speed, the volume of the grinding medium and fly ash in the tank expands. FIGS. 6 B and 6C provide a schematical view of this phenomenon. In particular, FIG. 6 B shows the volume of the bed at rest, i.e., where the RPM of the shaft and the arms equals zero. However, when the shaft is rotated at a
25 sufficient RPM, the volume of the medium and fly ash increases to a value slightly less than twice its volume when at rest. It has been discovered that unexpectedly, if grinding occurs at an RPM at which the volume of the grinding medium and fly ash essentially does not expand, then a greater amount of grinding medium can be added to the tank than the amount used in an expanded bed. This allows increases in the feed rate of fly ash to the
30 grinding process so that desirable rates of fly ash product are obtained, i.e. about 3 tons per hour. However, in order for the non-expanded bed process to be used, RPMs less than those used in the expanded bed process are employed. Therefore, the grinding medium in the non-expanded bed process must have a density greater than that of the grinding medium

used in an expanded bed process, so that the same ratio of fly ash to grinding medium, e.g., 1:4 by volume, and energy input can be used to grind the fly ash without expanded the bed.

- 5 As used herein, the term "fly ash" refers to a solid material having a chemical composition similar to or the same as the composition of the material that is produced during the combustion of powdered coal. In a specific aspect, the solid material is the material remaining after the combustion of powdered coal. ACI Committee 116 (1990, ACI 116-85, *ACI Manual of Concrete Practice Part I*, American Concrete Institute, Detroit) defines
- 10 fly ash as "the finely divided residue resulting from the combustion of ground or powder coal which is transported from the firebox through the flue gases", and the term "fly ash" as used herein encompasses this definition. Generally, fly ash derived from various coals have differences in chemical composition, but the principal components of fly ash are SiO_2 (25% to 60%), Al_2O_3 (10% to 30%), and Fe_2O_3 (5% to 25%). The MgO content of fly ash
- 15 is generally not greater than 5%. Thus, the term fly ash generally refers to solid powders comprising from about 25% to about 60% silica, from about 10% to about 30% Al_2O_3 , from about 5% to about 25% Fe_2O_3 , from about 0% to about 20% CaO, and from about 0% to about 5% MgO.
- 20 The term "fly ash" further contemplates synthetic fly ash, which may be prepared to have the same performance characteristics as fly ash as described herein.

- Presently, fly ash is classified primarily in two groups: Class C and Class F, according to the ASTM C 618 (1990, *supra*). Class F is generally produced by burning bituminous
- 25 coal, and Class C results from sub-bituminous coal or lignite. Generally, the fly ash from the combustion of sub-bituminous coals contains more CaO and less Fe_2O_3 than fly ash from bituminous coal (Berry and Malhotra, 1980, ACI J. Proceedings 77:59-73). Thus, the CaO content of the Class C fly ash is usually higher than 10%, with the sum of the oxides of SiO_2 , Al_2O_3 and Fe_2O_3 not less than 50%. For Class F fly ash the CaO content is
 - 30 normally less than 10% and the sum of the above mentioned oxides is not less than 70%.

The glassy phase of fly ash depends essentially on the combustion conditions and type of boiler. Non-fractionated fly ash obtained from different boilers, such as dry bottom boilers

or wet bottom boilers, has been found to behave differently. Boilers that achieve higher temperature yield fly ash with a more developed or pronounced glassy phase.

Alternatively, combustion in the presence of a fluxing agent, which reduces the fusion temperature of the fly ash, can also increase the glassy phase of fly ash produced by

5 combustion for lower temperature boilers. Compressive strength of a hardenable mixture containing fly ash may depend in part on the glassy phase of the fly ash, so generally fly ash produced for higher temperature boilers, or produced in the presence of a fluxing agent, or both, may be preferred. However, the fineness modulus is the most important parameter for defining compressive strength, and fractionated fly ash from any source, with
10 a defined fineness modulus, can be used according to the invention.

Although fly ash generally comes in a dry and finely divided form, in many instances, due to weathering and transportation processes, fly ash becomes wet and often forms lumps.

Such fly ash can be less reactive.

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Pozzolanic, as defined by ASTM C 593 (1990, ASTM C 593-89, *Annual Book of ASTM Standards*, Vol. 04.02), is "a siliceous or aluminosiliceous material that in itself possesses little or no cementitious value but that in finely divided form and in the presence of moisture will chemically react with alkali and alkaline earth hydroxides at ordinary

20 temperatures to form or assist in forming compounds possessing cementitious properties."

The present invention relates to the determination of the fineness modulus of fractionated fly ash. As used herein, the term "fineness modulus" refers to a measure of the distribution of volumes of particles of fly ash or distribution of particle sizes of the fly ash.

According to the present invention, the fineness modulus is a distribution analysis that is
25 much more informative than an average or median particle diameter determination or total surface area determination. The value of fineness modulus corresponds to the fineness of a fraction of fly ash, or to non-fractionated fly ash. Thus, a fraction of fly ash containing a distribution of particles having smaller size, *e.g.*, a median diameter that falls within a smaller range set, will have a fineness modulus value that is lower than a fraction of fly ash
30 containing a distribution of particles having somewhat larger size, *e.g.*, a median diameter that falls within a larger range set, or non-fractionated fly ash. According to the present invention, lower values of fineness modulus are preferred, since hardenable mixtures that contain fractions having a lower fineness modulus achieve compressive strength gains more

rapidly. In another embodiment, larger values of fineness modulus may be preferred, where a slower rate of compressive strength gain may be desired.

The fineness modulus can be determined experimentally, *e.g.*, by sequentially sieving fly ash particles on different sized sieves, or by light diffraction index analysis. The latter method provides a basis for virtual sieving by indicating the size distribution and number, thus allowing for prediction of percent retention (or pass through) on a sieve of a given size.

Thus, the present invention is directed, in part, to use of processed fly ash, in which the fly ash particles in any given fraction have a more uniform distribution of volumes, sizes, and regular spherical shape than non-fractionated fly ash, or fly ash processed by crushing or other techniques that fracture the particles, resulting in irregular, microscopically jagged shape.

Preferably, the fineness modulus is determined as the sum of the percentage of fly ash remaining on each of a series of different sized sieves. As noted above, this value can be a virtual number, determined by predicting the percent retention on different sized sieves from size information derived using other techniques. Accordingly, the term "fineness modulus" refers to a relative value, which can vary depending on the series of sieves (or other sizing apparatus) chosen. Since, according to the instant invention, fly ash particles of smaller size or diameter are preferred for use in hardenable mixtures, more accurate determinations of fineness modulus are available if a series of smaller sieves are chosen. Preferably, the size of the sieves is predominantly below 10 μ , *e.g.*, the sieves may be 0.5, 1, 2, 3, 4, 5, 6, 7, 8 and 10 microns, with sieves ranging up to 300 microns being useful. The number of sieves sized 10 microns or less should be at least one more than the number of sieves sized greater than 10 microns. In a preferred embodiment, the number of sieves sized 10 microns or less is at least five. Although in a specific embodiment dry sieves are used to calculate a value for the fineness modulus, other methods, such as wet sieving, can also be used. Quantitative determination of fineness modulus, as well as preparation of hardenable mixtures with greater rates of increase in compressive strength, is more fully developed in co-pending U.S. Patent 5,624,491, entitled "IMPROVED COMPRESSIVE STRENGTH OF CONCRETE AND MORTAR CONTAINING FLY ASH," and

International Patent Publication No. WO 95/32423, published November 30, 1995, of International Patent Application No. PCT/US95/06182, filed May 19, 1995, by Liskowitz et al. and having the same title, both of which are incorporated herein by reference in their entireties.

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The greater the number of sieves sized 10 microns or less, the greater the absolute value of fineness modulus. Accordingly, where sieves of 0.5, 1, 2, 3, 4, 5, 6, 7, 8, and 10 microns are used, the fineness modulus will be a higher absolute number, reflective of the greater degree of accuracy of determination of this value for the smaller diameter or smaller size fly ash particles.

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Although not intending to be bound by any particular theory or hypothesis, it is believed that dissolution of fly ash in a hardenable mixture, whereby the pozzolanic properties of the fly ash can contribute to compressive strength of the hardenable mixture, is acutely dependent on the size distribution of the fly ash to a certain minimum size. It has been further discovered that fly ash particles with a uniform, generally spherical, shape are preferred because they do not degrade the workability of cementitious mixtures. The data disclosed in the Examples, *infra*, support a conclusion that the fly ash contribution to compressive strength of a hardenable mixture depends on the distribution of particle volumes, or sizes. Above a minimum size, the contribution diminishes. Below this minimum size, strength of the concrete appears to be independent of size. Most surprising is the discovery that a regular, predominantly spherical, surface is preferred. This observation is surprising because the surface area hypothetically determines the reactivity of a particle, since surface functional groups are presumably more available for reaction, and thus one might expect that an irregular surface area, such as with a jagged particle, would be preferred.

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The pozzolanic reaction of fly ash in a hardenable mixture comprising cement is the reaction between constituents of the fly ash and calcium hydroxide or other alkaline materials. It is generally assumed to take place on the surface of fly ash particles, between silicates and aluminates from the glass phase of the fly ash and hydroxide ion in the pore solution (Plowman, 1984, Proceedings, 2nd International Conference on Ash Technology and Marketing, London, pp. 437-443). However, the result of the research leading to the

30

present invention indicates that the pozzolanic reactions of the ash are dependent on the volume and shape of the fly ash particles: the smaller the particle volume, and the more regular its shape, the more rapidly it completes its reaction with the cement to contribute to compressive strength. The rate of solubility and reactivity of these glassy phases in

5 different types of fly ash depends on the glassy phase of fly ash, which in turn depends on the combustion temperature of the boiler that produced the fly ash. In addition to the effect of combustion conditions on the glassy phase of fly ash, different fly ashes from one class can behave differently, depending on the SiO_2 , Al_2O_3 and Fe_2O_3 content, and other factors such as the particle size distribution and storage conditions of the ash (see Aitcin et al,

10 1986, *supra*; Liskowitz et al., 1983, *supra*).

During hydration, Portland cement produces a surfeit of lime (CaO) that is released to the pore spaces. It is the presence of this lime that allows the reaction between the silica components in fly ash and calcium hydroxide to form additional calcium silicate hydrate

15 [C-S-H]. He et al. (1984, Cement and Concrete Research 14:505-511) showed that the content of crystalline calcium hydroxide in the fly ash-Portland cement pastes decreases as a result of the addition of fly ash, most likely resulting from a reaction of calcium with alumina and silica from fly ash to form addition C-S-H. This process stabilizes the concrete, reduces permeability and increases resistance to chemical attacks.

20 Processing of fly ash to achieve the desired fineness modulus, *i.e.*, distribution of sizes, can be accomplished by any means known in the art, provided that it preserves or results in particles of fairly regular, preferably spherical, shape. In one example, processing comprises grinding with a fluidized bed grinding process with a low ratio of fly ash to

25 grinding media, such that the volume of fly ash is less than the void volume of the grinding medium. In a specific embodiment, *infra*, about one part unfractionated fly ash is ground with about seven parts grinding media (by weight), and the grinding media is zirconium silicate. However, other grinding media, such as carbon steel, are also contemplated for use according to the invention.

30 The term "cement" as used herein refers to a powder comprising alumina, silica, lime, iron oxide and magnesia burned together in a kiln and finely pulverized, which upon mixing with water binds or unites other materials present in the mixture in a hard mixture. Thus,

the hardenable mixtures of the invention comprise cement. Generally, the term cement refers to hydraulic cements such as, but not limited to, Portland cement, in particular Portland type I, II, III, IV and V cements.

- 5 As used herein, the term "cementitious materials" refers to the portion of a hardenable mixture that provides for binding or uniting the other materials present in the mixture, and thus includes cement and pozzolanic fly ash. Fly ash can comprise from about 5% to about 50% of the cementitious materials in a hardenable mixture of the invention; preferably, fly ash comprises from about 10% to about 35% of cementitious materials. The balance of
 10 cementitious materials will generally be cement, in particular Portland cement. In a specific embodiment, *infra*, the hardenable mixtures of the invention comprise Portland type I cement.

- The term "concrete" refers to a hardenable mixture comprising cementitious materials; a
 15 fine aggregate, such as sand; a coarse aggregate, such as but not limited to crushed basalt coarse aggregate; and water. Concrete of the invention further comprises fly ash having defined fineness. In a specific embodiment, the fly ash makes up from about 10% to about 50% of the cementitious materials. In a further aspect, the fly ash is used as fine aggregate in a ratio of from about 4:1 to about 1:1 to sand. In yet a further embodiment, the fly ash
 20 is an additive in addition to a replacement of cement, or a replacement of cement and fine aggregate.

- Various ratios of cement, fine aggregate, coarse aggregate, water, and optionally additives as set forth below, are used to prepare concrete, and are contemplated by the present
 25 invention. In specific embodiments, concrete of the invention consists essentially of about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, about 0.35 to about 0.6 parts by weight water, and optionally additives as set forth below, such that the ratio of cementitious materials to water ranges from approximately 3:1 to 1.5:1; preferably, the
 30 ratio of cementitious materials to water is about 2:1. In a specific embodiment, the concrete consists essentially of 1 part cementitious materials, 2 parts siliceous river sand or Ottawa sand, 3 parts 3/8" crushed basalt coarse aggregate, and 0.5 parts water.

The term "mortar" refers to a hardenable mixture comprising cementitious materials; a fine aggregate, such as sand; and water. Mortar of the invention further comprises fly ash having defined fineness. In a specific embodiment, the fly ash makes up from about 10% to about 50% of the cementitious materials. In a further aspect, the fly ash is used as fine aggregate in a ratio of from about 4:1 to about 1:1 to sand. In yet a further embodiment, the fly ash is an additive in addition to a replacement of cement, or a replacement of cement and fine aggregate.

Various ratios of cement, fine aggregate, water, and optionally additives as set forth below, are used to prepare mortar, and are contemplated by the present invention. In specific embodiments, mortar of the invention consists essentially of about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 0.5 parts by weight water, and optionally additives, such that the ratio of cementitious materials to water is approximately 2:1. In a specific embodiment, the mortar consists essentially of 1 part cementitious materials, 2.75 parts Ottawa sand, and 0.5 parts water.

As noted above, fly ash can be used as a fine aggregate in concrete or mortar, in addition to having a role as a cementitious material. It has been found that substituting fly ash for a conventional fine aggregate, such as sand, provides the advantages of increased compressive strength of the concrete or mortar since the total amount of fly ash in the hardenable composition is the same, with a rapid rate of increase of compressive strength because the amount of cement in the cementitious materials is greater.

According to the present invention, the hardenable mixture can further comprise one or more of the following in quantities commonly used in concrete and mortar compositions: kiln dust, *e.g.*, the dust generated in the manufacture of cement; silica fume, which is a by-product from the silicon metal industry usually consisting of about 96%-98% reactive SiO_2 , and which generally comes in very fine particle sizes of less than 1 micron; superplasticizer, such as Daracem-100 (W.R. Grace), an expensive but common additive for concrete used to decrease the water requirement for mixing the concrete; and a dispersing agent, such as sodium hexametaphosphate (NaPO_3). The use of a dispersing agent is particularly preferred when weathered fly ash is incorporated in the hardenable mixture.

Addition of silica fume can enhance the early rate of strength gain of a hardenable mixture, and therefore may be a desirable component of hardenable mixtures of the invention.

In a specific embodiment, a hardenable mixture of the invention may also contain glass
 5 fibers for reinforcement. The use of glass fibers in hardenable mixtures of the invention for reinforcement can be achieved because the fly ash, particularly finer fractions of fly ash, reacts more readily than glass fibers with reactive components of the cement, *e.g.*, Ca(OH)_2 and continues to react with an excess of fine fly ash particles made pursuant to the instant invention, thus preventing long term reaction of the glass fibers with these reactive
 10 components, which would otherwise degrade the glass fibers. The most inert hardenable mixtures are those that contain approximately equal amounts of fly ash, or fly ash and silica fume (as discussed below), and cement. The ability of fly ash to neutralize reactive agents in cement is discussed in greater detail in U.S. Application Serial No. 08/246,861, filed May 20, 1994, and International Patent Publication No. WO 95/32162, published
 15 November 30, 1995, of International Application No. PCT/US95/06336, filed May 19, 1995, both entitled "SULFATE AND ACID RESISTANT CONCRETE AND MORTAR" by John Liskowitz et al., which is incorporated herein by reference in its entirety.

In another specific embodiment, a hardenable mixture of the invention further comprises
 20 glass fibers, and silica fume. Silica fume reacts more readily with reactive components of cement than the glass fibers, and thus can provide early desirable protection of the glass fibers from degradation as well as early compressive strength gains. Subsequently, the fly ash will react with such reactive components, thus precluding early and late reactivity of glass fibers. As noted above, reaction of glass fibers with alkali and alkali earth
 25 compounds can lead to degradation of the glass fibers, and loss of tensile strength of the hardenable mixture.

Concrete beams of the invention with dimensions of 3"x6"x27" can be used to evaluate the bending strength of fly ash concrete, *e.g.*, using simple beam with third-point loading.
 30 Preferably, such test procedures are in accordance with ASTM C 78 (1990, ASTM C 78-84, *Annual Book of ASTM Standards*, Vol 04.02).

The present invention will be better understood by reference to the following Examples,

which are provided by way of exemplification and not by way of limitation. The Examples compare various techniques for processing fly ash, including air classification and grinding, and demonstrate that grinding a specific starting materials under appropriate conditions is necessary to produce a processed fly ash with the advantages disclosed and exemplified herein.

EXAMPLE 1: AIR CLASSIFICATION OF FLY ASH

Although it has been generally recognized that fly ash produced in utility coal fired boilers when used in small quantities ($\leq 15\%$) as a replacement for cement can provide desirable properties to cement products, its widespread use by the cement industry has been severely limited. This is due to variability in the properties of fly ash collected from different boilers at different times, its retarding early strength development, and lack of accurate relationship to predict performance of fly ash in cement products when the quantity of the fly ash used as cement replacement exceeds 15%. This occurs because of a lack of understanding of the factors and fly ash properties that influence the performance of the fly ash in combination with the cement in cement products. As a result, fly ash has been used in small amounts as a generic material in cement to gain improvement in the engineering properties of the cement products without affecting the performance of the cement products that can result from variability in the fly ash properties. The present Examples address these deficiencies, and demonstrates that quantitative utilization of fly ash can be achieved in high quality concrete and mortar compositions.

Materials and Methods

Twenty super sacs (10 tons total) of marketable fly ash were collected from an electricity generation boiler. An MS-20 commercial air classification fractionater (Progressive Industries, Sylacanga, Alabama) was employed to obtain the desired fly ash product. The coarse fraction of fly ash that is produced along with the desirable fly ash product in the fractionater was intended to be ground to produce a targeted ground fly ash particle size distribution that is comparable to the fractionated fly ash product.

Grinding of the coarse fraction from the MS-20 Progressive Industry Fractionater was carried out using a fluidized bed process (Union Processing, Akron, Ohio). As part of this

effort, grinding of the fly ash collected from the boiler was also investigated to determine if the desirable fly ash particle sizes in the fly ash could be increased with grinding. Also, dependence of desirable fly ash product on coal pulverizer performance and boiler operating conditions could be minimized through grinding. The amount of fly ash product with desirable particle sizes available in the fly ash was averaging only about 55 percent of the total particles present in the samples that were collected and analyzed without further processing.

Results and Discussion

Twelve different MS-20 fractionation control settings were evaluated for their effects on product yield. The processing rate was adjusted in the different settings. Increasing the processing rate of the MS-20 from 3 tons/hour up to 5 tons/hr reduced the product yield from 38% down to 28%, respectively. However, particle size analysis using the Leeds and Northrup Microtrac shows that the fly ash product produced at the greater processing rate of 5 tons/hr (M8F95) contained a greater fraction of fine particle sizes than the product produced at a rate of 3 tons/hour (M9F95). Selecting 11 microns as the desired maximum particle size (diameter) in the product, and 5.5 microns as the mean particle size range for comparison of size distribution, 93% and 78% of the particles in the M8F95 product were less than 11 microns and 5.5 microns, respectively (see Table 1), whereas 92% and 73% of the particles in the M9F95 are seen to be less than 11 and 5.5 microns (see Table 2). The median particle diameter for the M8F95 and M9F95 fractions was measured to be 2.8 microns and 3.4 microns, respectively.

Table 1

MICROTRAC STANDARD RANGE PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS			
5	Id #1: M8F95 Distrib. Format: Volume Filter: n/a Run Time: 15 seconds	Summary Data dv = 0.0579 10% = 1.121 50% = 2.828	Chan. Progression: Standard Upper Channel Edge: 704.0 Lower Channel Edge: 0,688 Number of Channels: 20
10	Run Number: Avg of 3 runs Transmission: 0.87 Laser Int: 1,018/0.000/0.000 Residuals: Disabled Above Residual: 0.000 Below Residual: 0.000	90% = 8.924 mv = 4.228 mn = 1.217 ma = 2.306 ca = 2.602 sd = 2.656	
15			
	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	497.8	100.00	0.00
	352.0	100.00	0.00
20	248.9	100.00	0.00
	176.0	100.00	0.00
	124.5	100.00	0.00
	88.00	100.00	0.00
	62.23	100.00	0.00
25	44.00	100.00	0.03
	31.11	99.97	0.83
	22.00	99.14	2.37
	15.56	96.77	3.78
	11.00	92.99	5.45
30	7.778	87.54	9.13
	5.500	78.41	15.46
	3.889	62.95	14.13
	2.750	48.82	16.66
	1.945	32.16	14.54
35	1.375	17.62	11.94
	0.972	5.68	5.68

Table 2

MICROTRAC STANDARD RANGE PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
5	Id #1: M9F95 Distrib. Format: Volume Filter: n/a Run Time: 15 seconds Run Number: Avg of 3 runs Transmission: 0.93 Laser Int: 1,016/0.000/0.000 Residuals: Disabled Above Residual: 0.000 Below Residual: 0.000	Summary Data dv = 0.0316 10% = 1.153 50% = 3.357 90% = 10.06 mv = 4.710 mn = 1.220 ma = 2.480 ca = 2.420 sd = 3.130
10		Chan. Progression: Standard Upper Channel Edge: 704.0 Lower Channel Edge: 0,688 Number of Channels: 20
15		
20	Ch Top 704.0 497.8 352.0 248.9 176.0 124.5 88.00 62.23 44.00 31.11 22.00 15.56 11.00 7.778 5.500 3.889 2.750 1.945 1.375 0.972	% Pass 100.00 100.00 100.00 100.00 100.00 100.00 100.00 100.00 100.00 99.95 98.84 96.12 91.53 84.49 73.16 55.70 43.03 29.13 16.03 5.19
25		% Change 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.05 1.11 2.72 4.59 7.04 11.33 17.46 12.67 13.90 13.10 10.84 5.19
30		
35		

- 40 A comparison of the compressive strength development exhibited by the M8F95 and
M9F95 fly ash-containing mortar in mortar with 25% cement replacement achieved after up
to twenty-eight days of curing show differences between the two products. Comparing
Figure 1 (M8F95-25) with Figure 2 (M9F95-25), the compressive strength development
achieved with the M8F95-25 mortar is seen to be slightly greater than that achieved with
45 M9F95-25 mortar up to fourteen days of curing. After twenty eight days of curing, both

the M8F95-25 and M9F95-25 mortar exhibit comparable compressive strength.

Apparently, the finer particle size distribution present in the M8F95 product compared to the M9F95 particle size distribution accounts for faster early compressive strength development exhibited by the M8F95-25 mortar.

5

Mortars were also prepared with M8F95 and M9F95 fly ash products by replacing 35% of the cement with these fly ash products. These mortars did not have observable differences in the rate or extent of compressive strength (compare Figure 3, M8F95-25, with Figure 4, M9F95-35) between them. The use of larger amounts of fly ash in the mortar apparently masks the influence of differences in particle size distribution in the M8F95 and M9F95 products on compressive strength development.

10

Compressive strength development exhibited by the mortars prepared with 25% replacement of the cement with the M9F95 fly ash product and the M13N94 fly ash product prepared from ash collected in late 1994 provided comparable results. If one compares Figure 2 with Figure 5, the compressive strengths measured at seven days, fourteen days and twenty eight days of curing can be seen to be comparable for the M9F95-25 and M13N94-25 mortars, respectively.

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This result was consistent with our predictions from the fineness modulus, since the control setting on the fractionator used to prepare the M13N94 ash provide a processing rate of 3.2 ton/hour which was comparable to the 3 ton/hour processing rate used for the M9F95 product. Particle size analysis indicated their particle size distributions are similar (compare Table 2 with Table 3).

20

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Table 3

MICROTRAC STANDARD RANGE PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
5		
10	Id #1: M13N Distrib. Format: Volume Filter: n/a Run Time: 15 seconds Run Number: Avg of 3 runs Transmission: 0.92 Laser Int: 1,006/0.000/0.000 Residuals: Disabled Above Residual: 0.000 Below Residual: 0.000	Summary Data dv = 0.0392 10% = 1.198 50% = 3.943 90% = 8.718 mv = 4.557 mn = 1.235 ma = 2.638 ca = 2.275 sd = 2.961
15	Chan. Progression: Standard Upper Channel Edge: 704.0 Lower Channel Edge: 0,688 Number of Channels: 20	
20	<u>Ch Top</u> 704.0 497.8 352.0 248.9 176.0 124.5 88.00 62.23 44.00 31.11 22.00 15.56 11.00 7.778 5.500 3.889 2.750 1.945 1.375 0.972	<u>% Pass</u> 100.00 100.00 100.00 100.00 100.00 100.00 100.00 100.00 100.00 100.00 99.87 98.06 95.54 86.09 69.90 49.29 37.70 25.96 14.27 4.61
25	<u>% Change</u> 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.13 1.81 2.52 9.45 16.19 20.61 11.59 11.74 11.69 9.66 4.61	
30		
35		

40 These results indicate that proper sittings of the MS-20 fractionater at given feed rates to achieve specific yields of fly ash product can minimize variability in fly ash pozzolanic activity that may exist in fly ash samples collected at different times or from different boilers. Increasing the feed rate increases the quantity of fine particle sizes in the product but at the expense of the product yield.

45

EXAMPLE 2: GRINDING RESULTS

The coarse fraction (M8CG) produced along with the M8F95 fly ash product in the MS-20 fractionator was selected for grinding. The M8CG fraction was chosen over the M9CG fraction because particle size analysis of these two coarse fractions reveal that 47% of the M8CG fraction particles were below 11 microns and 37% of the M9CG fraction were below 11 microns. Fly ash particle sizes 11 microns and below are the preferred particle sizes for fly ash products to be incorporated in concrete or mortar. Both the grinding of the M8CG coarse fraction and the unfractionated fly ash was investigated using the Union Process Dry Grinding Attritor whose principle of operation is described in Figure 6 (prior art). This grinding process was selected because it was assumed that grinding and rotation of the fly ash particles in an expanded media bed would produce ground particles that are similar in shape to the spherical unground particle. The spherical shape of fly ash particles in cement improves the workability of the fly ash cement product.

A series of six grinding experiments were carried out in a HSA-30 Attritor, which allows scale-up and assessment of the grinding performance of the commercial size Attritor without the need for large quantities of sample. The grinding experiments were carried out by changing the quantity of 2-2.5 mm zirconium silicate beads which served as the grinding media in the Attritor, and the feed rate. The grinding and compressive strength development achieved with the ground M8CG2 product produced from the coarse M8CG fraction and the ground product (M0G95) prepared from the unfractionated fly ash were evaluated.

Microscopic examination of the ground fly ash particles indicates uniformity in shape, comparable to the spherical fractionated fly ash product produced by the air classification process. These results indicate that the Attritor grinding process achieves size reduction by surface grinding as opposed to fracturing the fly ash particles. If fracturing occurred, one would expect to observe irregular shaped particles.

Comparison of the results of the grinding of the M8CG coarse fraction and the unfractionated fly ash shows that the ground products obtained from the unfractionated fly ash (M0G95) is finer than the ground M8CG2 fraction. In this experiment, we found that

90% of the ground M0G95 fly ash product is smaller than 11 microns, and that 73% is smaller than 5.5 microns, respectively (see Table 4), with the median particle size measured to be 3.0 microns. The percentage of the particle sizes below 11 microns and 5.5 microns measured in the ground M8CG2 product was 89% and 58%, with a median diameter of 4.6 microns (see Table 5).

Table 4

MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
10	Id #1: FLY ASH RAW FEED 2 Distrib. Format: Volume Filter: On Run Time: 15 seconds Run Number: Avg of 2 runs Transmission: 0.87 Laser Int: 1,007/0.005/0.999 Residuals: Disabled Above Residual: 0.000 Below Residual: 0.000	Summary Data dv = 0.0518 10% = 0.827 50% = 2.992 90% = 10.75 mv = 21.74 mn = 0.514 ma = 1.820 ca = 3.297 sd = 3.369
15		Chan. Progression: Standard Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 50
20		

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	592.0	100.00	0.00
	497.8	100.00	0.00
5	418.6	100.00	1.31
	352.0	98.69	2.96
	296.0	95.73	1.32
	248.9	94.41	0.00
	209.3	94.41	0.00
10	176.0	94.41	0.00
	148.0	94.41	0.00
	124.5	94.41	0.00
	104.7	94.41	0.00
	88.00	94.41	0.00
15	74.00	94.41	0.00
	62.23	94.41	0.00
	52.33	94.41	0.00
	44.00	94.41	0.00
	37.00	94.41	0.00
20	31.11	94.41	0.00
	26.16	94.41	0.15
	22.00	94.26	0.45
	18.50	93.81	0.72
	15.56	93.09	1.10
25	13.08	91.99	1.70
	11.00	90.29	2.56
	9.250	87.73	3.71
	7.778	84.02	4.96
	6.541	79.06	6.02
30	5.500	73.04	6.63
	4.625	66.41	6.76
	3.889	59.65	6.51
	3.270	53.14	5.97
	2.750	47.17	5.39
35	2.312	41.78	5.08
	1.945	36.70	5.21
	1.635	31.49	5.71
	1.375	25.78	6.06
	1.156	19.72	5.60
40	0.972	14.12	4.35
	0.818	9.77	3.00
	0.688	6.77	2.03
	0.578	4.74	1.43
	0.486	3.31	1.08
45	0.409	2.23	0.87
	0.344	1.36	0.73
	0.289	0.63	0.63
	0.243	0.00	0.00
	0.204	0.00	0.00
50	0.172	0.00	0.00
	0.145	0.00	0.00

Table 5

5	MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
10	Id #1: FLY ASH 8 COARSE Distrib. Format: Volume Filter: On Run Time: 15 seconds Run Number: Avg of 2 runs Transmission: 0.94 Laser Int: 1,007/0.005/0.999 Residuals: Disabled Above Residual: 0.000 Below Residual: 0.000	Summary Data dv = 0.0291 10% = 0.947 50% = 4.577 90% = 11.40 mv = 5,570 mn = 0.479 ma = 2.265 ca = 2.649 sd = 4.093	Time: 10:30 Chan. Progression: Standard Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 50
15			

60440 "044400

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	592.0	100.00	0.00
	497.8	100.00	0.00
5	418.6	100.00	0.00
	352.0	100.00	0.00
	296.0	100.00	0.00
	248.9	100.00	0.00
	209.3	100.00	0.00
10	176.0	100.00	0.00
	148.0	100.00	0.00
	124.5	100.00	0.00
	104.7	100.00	0.00
	88.00	100.00	0.00
15	74.00	100.00	0.00
	62.23	100.00	0.00
	52.33	100.00	0.00
	44.00	100.00	0.00
	37.00	100.00	0.00
20	31.11	100.00	0.39
	26.16	99.61	0.66
	22.00	98.95	1.10
	18.50	97.85	1.81
	15.56	96.04	2.81
25	13.08	93.23	4.20
	11.00	89.03	5.88
	9.250	83.15	7.53
	7.778	75.62	8.55
	6.541	67.07	8.64
30	5.500	58.43	7.98
	4.625	50.45	7.02
	3.889	43.43	6.02
	3.270	37.41	5.04
	2.750	32.37	4.20
35	2.312	28.17	3.63
	1.945	24.54	3.44
	1.635	21.10	3.52
	1.375	17.58	3.65
	1.156	13.93	3.46
40	0.972	10.47	2.85
	0.818	7.62	2.10
	0.688	5.52	1.50
	0.578	4.02	1.10
	0.486	2.92	0.87
45	0.409	2.05	0.74
	0.344	1.31	0.68
	0.289	0.63	0.63
	0.243	0.00	0.00
	0.204	0.00	0.00
50	0.172	0.00	0.00
	0.145	0.00	0.00

Differences in particle size distribution between the ground M8CG2 product and the ground
55 M0G95 product under the same specific grinding operating conditions using a ratio of one
part fly ash to seven parts grinding media (by weight) appear to be due to the differences in
particle size distribution exhibited by the pre-ground M8CG feed and pre-ground fly ash

feed. A comparison of particle size distributions show that the particle sizes in the M8CG coarse fraction (see Figure 7) is skewed toward the 5 micron to 88 micron range. The unground fly ash contains a broad distribution of sizes, primarily in 0.9 micron to 62 micron range (see Figure 8). The grinding of the two fly ash samples with different particle size distributions resulted in increasing the quantity of particles with sizes below 1.375 and 5.5 microns in the M8CG sample from 6% to 18% and 18% to 58%, respectively, and in the unground fly ash from 14% to 26% and 40% to 73%, respectively.

- 10 These data show that direct grinding of the fly ash eliminates the need for initial fractionation of the fly ash followed by grinding of the coarse product to improve the yield. A comparison of the particle size distribution of the M8F95 product produced with the MS-20 fractionation and the M0G95 product achieved with direct grinding of the fly ash suggests that they are similar. The percentage of particle sizes below 11 microns, 5.5
- 15 microns, and 1.375 microns in the M8F95 product are 93%, 78%, and 18%, respectively, with a median particle diameter of a 2.8 microns (see Table 1). The percentages for the particle sizes below 11 microns, 5.5 microns, and 1.375 microns in the ground M0G95 product are 90%, 73% and 26%, respectively, with a median diameter of 3 microns (see Table 4).

- 20 A comparison of the compressive strength development of mortar prepared with 25 percent replacement of cement with the ground M8CG2 and ground M0G95 fly ash shows the ground unfractionated fly ash mortar (M0G95-25) exhibits a faster rate of compressive strength gain, and overall greater compressive strength gain, than the ground course fraction (M8CG2-25)-containing mortar up to the twenty eight day design strength. This is
- 25 observed by comparison of compressive strength development of the M0G95-25 mortar with curing age in Figure 9 with that measured for the M8CG-2-25 mortar in Figure 10. These results are consistent with our expectations based on comparison of the quantity of fine particle sizes in the ground M0G95 product, which is greater than in the ground
- 30 M8CG2 product.

Compressive strength development of mortars, M0G95-25, M0G95-35, M8F95-25, and M8F95-35, prepared with the ground M0G95 unfractionated fly ash product and the

fractionated product (M8F95) using 25% and 35% cement replacement with the fly ash products are comparable. This is seen by comparing the compressive strength development for M8F95-25 mortar in Figure 1 with compressive strength development for M0G95-25 in Figure 9 and compressive strength development M8F95-35 in Figure 3 with that for M0G95-35 in Figure 11.

The compressive strength development of the mortars M8F95-25 and M0G95-25, prepared with 25 percent replacement of cement with the fractionated M8F95 product or ground unfractionated M0G95 product, achieves control strength between 7 and 14 days of curing (compare Figure 12 with Figure 1 and Figure 9, respectively). The compressive strength of the M8F95-35 and M0G95-35 mortars prepared with 35 percent placement of cement with the M8F95 and M0G95 fly ash product achieves control strength at 14 days of curing (compare Figure 12 with Figure 3 and Figure 11, respectively).

Conclusions

Particle surface grinding achieved with the Union Process Attritor provided targeted fly ash particle size distribution and particle shapes that are comparable to the desirable fly ash product produced with the air classification process. Compressive strength development achieved with mortar up to the 28 day design strength using ground unfractionated Mercer fly ash as a cement replacement is comparable to that achieved with the fly ash product produced in the commercial size Progressive Industries MS-20 air classifier. Compressive strength development exhibited by mortar prepared with 25% cement replacement with ground unfractionated fly ash exceeds control compressive strength between 7 and 14 days of curing.

Most surprisingly, the grinding process produces a desired fly ash product with 100% product yield without the need for air classification. The use of the grinding process can minimize the dependence of produce yield on operating conditions employed in conventional coal fired utility boilers.

The fly ash product has been demonstrated to significantly enhance the strength development and durability of concrete. The two major distinct characteristics of the fly ash product are (1) it performs as good as or better than normal cement using the same

design mix portions at early age; and (2) it exhibits exceptional resistance against acid and chemical attack. The studies carried out so far utilized conventional mix proportions as recommended by ASTM. No additives or admixtures were added in the tested mix proportions since the studies were aimed at evaluating and understanding the performance characteristics of the fly ash product. These data point the way for developing commercial formulations of fly ash product mortar and concrete for permeability, freeze-thaw resistance, mortar and concrete with early strength, mortar and concrete with strong acid and salt resistance for repair applications, concrete for glass fiber reinforced composites, and concrete with fly ash product and microsilica (silica fume).

EXAMPLE 3: GRINDING RESULTS USING A NON-EXPANDED BED

In this example, both an expanded and non-expanded bed were used to grind fly ash. In particular, an unprocessed, raw fly ash was selected for grinding. The grinding of the raw fly ash in a non-expanded bed was investigated using the Union Process Dry Grinding Attritor whose principle of operation is described in Figure 6.

Two experiments comprising a series of eleven grinding runs were carried out in a HSA-30 Attritor, which allows scale-up and assessment of the grinding performance of the commercial size Attritor without the need for large quantities of sample. The first three grinding runs involved an expanded bed, wherein the shaft of the grinder had a speed of 600 RPM. The remaining grinding runs were performed with a non-expanded bed, wherein the speed of the shaft was 400 RPM, with a current of 65 amps. In order to maintain this amperage in the non-expanded bed, i.e., transfer the same amount of energy to the grinding medium, an additional 5.8 gallons of grinding medium were used in the non-expanded bed experiments. The grinding medium used in these experiments was carbon steel spheres having a diameter of about 1/8 inch, and a packing density of about 40.2 lbs/gallon.

Experiment 1: Expanded Bed grinding

In the first experiment three runs were made grinding fly ash in an expanded bed.

Run No. 1

- Machine: HSA-30
 Tank: SS
 Arms: 8 L
 Diverter Discs: 3
 5 Tank Volume: 28.6 gallons
 Cooling Water: Yes
 Discharge Screen: 0.8 MM/LD
 Open Area: 1-Side 100%
 Horse Power: 50
 10 RPM Setting: 600
 Power: 65 amps
 Original Particle Size: DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
 Desired Particle Size: 90% - 12 μm
 Process Procedure: Continuous Dry Grind
 15 Test Data: 1) Material Processed: 180 lbs/10 minutes
 2) Product Temperature: 260° F
 3) Cooling Water Inlet temperature: 55° F / Outlet: 82° F
 4) Cooling Rate: 10 GPM
 5) Feeder Setting: 460
 20 Process Rate: 1080 lbs/hour

The grinding medium comprised 440 lbs (5.7 gallons) of carbon steel spheres with a diameter of about 1/8 inch.

- 25 The Results of the grinding are set forth below in Table 6:

Table 6

MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
30	Id #1: FLY ASH 8 COARSE Distrib. Format: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 2 runs Transmission: 0.94 Laser Int: 1,008/1.002/1.004 Residuals: Disabled Above Residual: 0.000 Below Residual: 0.000	Summary Data dv = 0.0356 10% = 2.030 50% = 5.156 90% = 22.21 mv = 30.38 mn = 1.870 ma = 4.180 cs = 1.436 sd = 5.591
35		Id # 2: Fly ash Low Load Time: 11:00 Chan. Progression: Geom/8rt2 Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp. Spherical Particles: No Part. Refractive Index: 1.81
40		

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
5	592.0	100.00	0.00
	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.06
10	383.9	99.94	0.43
	352.0	99.51	1.76
	322.8	97.75	2.21
	296.0	95.54	1.86
	271.4	93.68	1.31
	248.9	92.37	0.45
15	228.2	91.92	0.11
	209.3	91.81	0.00
	191.9	91.81	0.00
	176.0	91.81	0.00
20	161.4	91.81	0.00
	148.0	91.81	0.00
	135.7	91.81	0.00
	124.5	91.81	0.00
	114.1	91.81	0.00
25	104.7	91.81	0.00
	95.96	91.81	0.00
	88.00	91.81	0.00
	80.70	91.81	0.00
	74.00	91.81	0.00
30	67.86	91.81	0.00
	62.23	91.81	0.00
	57.06	91.81	0.00
	52.33	91.81	0.00
	47.98	91.81	0.00
	44.00	91.81	0.00
35	40.35	91.81	0.00
	37.00	91.81	0.13
	33.93	91.68	0.20
	31.11	91.48	0.25
40	28.53	91.23	0.33
	26.16	90.90	0.42
	23.99	90.48	0.54
	22.00	89.94	0.67
	20.17	89.27	0.83
45	18.50	88.44	1.01
	16.96	87.43	1.20
	15.56	86.02	1.42
	14.27	84.81	1.65
	13.08	83.16	1.90
	12.00	81.62	2.15
50	11.00	79.11	2.41
	10.09	76.70	2.68
	9.250	74.02	2.94
	8.482	17.08	3.19
	7.778	67.89	3.42
55	7.133	64.47	3.63
	6.541	60.84	3.81
	5.998	57.03	3.97
	5.500	53.06	4.09
	5.044	48.97	4.19
60	4.625	44.78	4.25
		4	

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Chn</u>
5	4.241	40.53	4.28
	3.889	36.25	4.26
	3.566	31.99	4.15
	3.270	27.84	3.97
	2.999	23.87	3.71
	2.750	20.16	3.36
10	2.522	16.80	3.00
	2.312	13.80	2.62
	2.121	11.18	2.26
	1.945	8.92	1.93
	1.783	6.99	1.64
15	1.635	5.35	1.37
	1.499	3.98	1.13
	1.375	2.85	0.90
	1.261	1.95	0.70
	1.156	1.25	0.50
20	1.060	0.75	0.36
	0.972	0.93	0.25
	0.892	0.14	0.14
	0.818	0.00	0.00
	0.750	0.00	0.00
25	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
	0.530	0.00	0.00
	0.486	0.00	0.00
30	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
	0.344	0.00	0.00
	0.315	0.00	0.00
35	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
	0.223	0.00	0.00
	0.204	0.00	0.00
40	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
	0.145	0.00	0.00
	0.133	0.00	0.00
45			

The data of Table 6 are graphically shown in Figure 13. The final particle size achieved in this experiment were:

90% of the particles had a size less than 22.21 μm ;

81.2 % of the particles had a size less than 12.0 μm ;

50 % of the particles had a size less than 5.15 μm ; and

the median value of the size of the particles was 30.83 μm

Run No. 2

A second experiment using an expanded bed was performed, and the following parameters were used:

- Machine: HSA-30
- 5 Tank: SS
- Arms: 8 L
- Diverter Discs: 3
- Tank Volume: 28.6 gallons
- Cooling Water: Yes
- 10 Discharge Screen: 0.8 MM/LD
- Open Area: 1-Side 100 %
- Horse Power: 50
- RPM Setting: 600
- Power: 65 amps
- 15 Original Particle Size: DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
- Desired Particle Size: 90 % - 12 μm
- Process Procedure: Continuous Dry Grind
- Test Data:
- 20 1) Material Processed: 239 lbs/10 minutes
- 2) Product Temperature: 230° F
- 3) Cooling Water Inlet temperature: 30° F / Outlet: 48° F
- 4) Cooling Rate: 10 GPM
- 5) Feeder Setting: 550
- Process Rate: 1434 lbs/hour
- 25 The grinding medium comprised 440 lbs (5.7 gallons) of carbon steel spheres with a diameter of about 1/8 inch. The Results of the grinding are set forth below in Table 7, and graphically set forth in Figure 14.

Table 7

30	MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
35	Id #1: FLY ASH 8 COARSE Distrib. Format: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 2 runs Transmission: 0.91 40 Laser Int: 1,008/1.002/1.004 Residuals: Disabled Above Residual: 0.000 Below Residual: 0.000	Summary Data dv = 0.0576 10% = 2.308 50% = 6.719 90% = 21.41 mv = 9.881 mn = 1.976 ma = 4.933 cs = 1.216 sd = 7.223	Id # 2: Fly Ash Med Load Time: 12:40 Chan. Progression: Geom/8rt2 Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp. Spherical Particles: No Part. Refractive Index: 1.81

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
	592.0	100.00	0.00
5	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.00
	383.9	100.0	0.00
10	352.0	100.0	0.00
	322.8	100.0	0.00
	296.0	100.0	0.00
	271.4	100.0	0.00
	248.9	100.0	0.00
15	228.2	100.0	0.00
	209.3	100.0	0.00
	191.9	100.0	0.00
	176.0	100.0	0.00
	161.4	100.0	0.00
20	148.0	100.0	0.00
	135.7	100.0	0.00
	124.5	100.0	0.00
	114.1	100.0	0.00
	104.7	100.0	0.00
25	95.96	100.0	0.00
	88.00	100.0	0.00
	80.70	100.0	0.00
	74.00	100.0	0.08
	67.86	99.92	0.13
30	62.23	99.79	0.19
	57.06	99.60	0.23
	52.33	99.37	0.26
	47.98	99.11	0.31
	44.00	98.80	0.38
35	40.35	98.42	0.48
	37.00	97.94	0.62
	33.93	97.32	0.80
	31.11	96.52	1.03
	28.53	95.49	1.29
40	26.16	94.20	1.61
	23.99	92.59	1.91
	22.00	90.68	2.21
	20.17	88.47	2.45
	18.50	86.02	2.64
45	16.96	83.38	2.76
	15.56	80.62	2.83
	14.27	77.79	2.88
	13.08	74.91	2.92
	12.00	71.99	2.97
50	11.00	69.02	3.06
	10.09	65.96	3.16
	9.250	62.80	3.30
	8.482	59.50	3.43
	7.778	56.07	3.56
55	7.133	52.51	3.64
	6.541	48.87	3.69

	5.998	45.18	3.70
	5.500	41.48	3.69
	5.044	37.79	3.64
	4.625	34.15	3.57
5	4.241	30.58	3.48
	3.889	27.10	3.37
	3.566	23.73	3.21
	3.270	20.52	3.02
	2.999	17.50	2.77
10	2.750	14.73	2.49
	2.522	12.24	2.20
	2.312	10.04	1.91
	2.121	8.13	1.64
	1.945	6.49	1.41
15	1.783	5.08	1.19
	1.635	3.89	1.01
	1.499	2.88	0.84
	1.375	2.04	0.68
	1.261	1.36	0.53
20	1.156	0.83	0.40
	1.060	0.43	0.27
	0.972	0.16	0.12
	0.892	0.04	0.04
	0.818	0.00	0.00
25	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
	0.530	0.00	0.00
30	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
	0.344	0.00	0.00
35	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
	0.223	0.00	0.00
40	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
	0.145	0.00	0.00
45	0.133	0.00	0.00

The final particle size achieved in this second expanded bed experiment were:

90 % of the particles had a size less than 21.41 μm ;

71.9 % of the particles had a size less than 12.0 μm ;

50 % of the particles had a size less than 6.71 μm ; and

the median value of the size of the particles was 9.88

Run No. 3

In this run, a third expanded grinding bed was used to grind fly ash, with the objective of

- 5 obtaining a final size of greater than 90% of the fly ash particles being 12 μm or less. The parameters used in the grinding are as follows:

	Machine:	HSA-30
	Tank:	SS
	Arms:	8 L
10	Diverter Discs:	3
	Tank Volume:	28.6 gallons
	Cooling Water:	Yes
	Discharge Screen:	0.8 MM/LD
	Open Area:	1-Side 100%
15	Horse Power:	50
	RPM Setting:	600
	Power:	65 amps
	Original Particle Size:	DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
	Desired Particle Size:	90% - 12 μm
20	Process Procedure:	Continuous Dry Grind
	Test Data:	1) Material Processed: 300 lbs/10 minutes
		2) Product Temperature: 210° F
		3) Cooling Water Inlet temperature: 28° F / Outlet: 48° F
		4) Cooling Rate: 10 GPM
25		5) Feeder Setting: 640
	Process Rate:	1434 lbs/hour

Hence, the amount of fly ash ground is increased relative to the amount ground in Experiments 1 and 2 above.

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The grinding medium comprised 440 lbs (5.7 gallons) of carbon steel spheres with a diameter of about 1/8 inch. The Results of the grinding are set forth below in Table 8, and graphically set forth in Figure 15.

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	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
	592.0	100.00	0.00
5	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.00
	383.9	100.00	0.00
10	352.0	100.00	0.00
	322.8	100.00	0.00
	296.0	100.00	0.00
	271.4	100.00	0.00
	248.9	100.00	0.00
15	228.2	100.00	0.00
	209.3	100.00	0.00
	191.9	100.00	0.00
	176.0	100.00	0.00
	161.4	100.00	0.00
20	148.0	100.00	0.00
	135.7	100.00	0.00
	124.5	100.00	0.00
	114.1	100.00	0.00
	104.7	100.00	0.08
25	95.96	99.92	0.11
	88.00	99.81	0.09
	80.70	99.72	0.13
	74.00	99.59	0.21
	67.86	99.38	0.27
30	62.23	99.11	0.28
	57.06	98.83	0.33
	52.33	98.50	0.39
	47.98	98.11	0.47
	44.00	97.64	0.59
35	40.35	97.05	0.75
	37.00	96.30	0.95
	33.93	95.35	1.20
	31.11	94.15	1.50
	28.53	92.65	1.81
40	26.16	90.84	2.16
	23.99	88.68	2.45
	22.00	96.23	2.69
	20.17	83.54	2.86
	18.50	80.68	2.94
45	16.96	77.74	2.97
	15.56	74.77	2.94
	14.27	71.83	2.91
	13.08	68.92	2.89
	12.00	66.03	2.92
50	11.00	63.11	2.68
	10.09	60.13	3.06
	9.250	57.07	3.18
	8.482	53.89	3.30
	7.778	50.59	3.39
55	7.133	47.20	3.47
	6.541	43.73	3.50
60			

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	5.998	40.23	3.49
	5.500	36.74	3.40
	5.044	33.34	3.32
	4.625	30.02	3.22
5	4.241	26.80	3.11
	3.889	23.69	2.98
	3.566	20.71	2.82
	3.270	17.89	2.63
	2.999	15.26	2.41
10	2.750	12.85	2.16
	2.522	10.69	1.90
	2.312	8.79	1.65
	2.121	7.14	1.43
	1.945	5.71	1.22
15	1.783	4.49	1.04
	1.635	3.45	0.88
	1.499	2.57	0.74
	1.375	1.83	0.60
	1.261	1.23	0.47
20	1.156	0.76	0.37
	1.060	0.39	0.24
	0.972	0.15	0.11
	0.892	0.04	0.04
	0.818	0.00	0.00
25	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
	0.530	0.00	0.00
30	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
	0.344	0.00	0.00
35	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
	0.223	0.00	0.00
40	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
	0.145	0.00	0.00
45	0.133	0.00	0.00

The final particle size range achieved in this third expanded bed experiment were:

90 % of the particles had a size less than 25.26 μm ;

66 % of the particles had a size less than 12 μm ;

50 % of the particles had a size less than 7.66 μm ; and

the median value of the size of the particles was 11.69

Thus, in these expanded bed grinding runs set forth in this Example, the desired final particle size of greater than 90 % of the particles having a size of 12 μm was not obtained. Furthermore, as the volume of fly ash ground increases, i.e, as the ratio of the volume of fly ash to the void volume decreased, the efficiency of the grinding process decreased.

5

Experiment 4: Grinding in a non-expanded bed

In this experiment, 8 runs were made using a non-expanded bed of the invention to grind fly ash. The volume of fly ash ground in each run was increased to the volume ground in the previous run.

10

Run No. 1

	Machine:	HSA-30
	Tank:	SS
	Arms:	8 L
15	Diverter Discs:	3
	Tank Volume:	28.6 gallons
	Cooling Water:	Yes
	Discharge Screen:	0.8 MM/LD
	Open Area:	1-Side 100%
20	Horse Power:	50
	RPM Setting:	400
	Power:	66 amps
	Original Particle Size:	DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
	Desired Particle Size:	90% - 12 μm
25	Process Procedure:	Continuous Dry Grind
	Test Data:	1) Material Processed: 270 lbs/15 minutes
		2) Product Temperature: 260° F
		3) Cooling Water Inlet temperature: 44° F / Outlet: 62° F
		4) Cooling Rate: 10 GPM
30		5) Feeder Setting: 440
	Process Rate:	1080 lbs/hour

The grinding medium comprises 11.5 gallons (462 lbs) of carbon steel spheres having a size of about 1/8 inch. The results obtained from the first run are set forth in Table 9, and graphically in

35 Figure 16.

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Year	1980	1981	1982	1983	1984	1985	1986	1987	1988	1989	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032	2033	2034	2035	2036	2037	2038	2039	2040	2041	2042	2043	2044	2045	2046	2047	2048	2049	2050	2051	2052	2053	2054	2055	2056	2057	2058	2059	2060	2061	2062	2063	2064	2065	2066	2067	2068	2069	2070	2071	2072	2073	2074	2075	2076	2077	2078	2079	2080	2081	2082	2083	2084	2085	2086	2087	2088	2089	2090	2091	2092	2093	2094	2095	2096	2097	2098	2099	2100
1980	1980	1981	1982	1983	1984	1985	1986	1987	1988	1989	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032	2033	2034	2035	2036	2037	2038	2039	2040	2041	2042	2043	2044	2045	2046	2047	2048	2049	2050	2051	2052	2053	2054	2055	2056	2057	2058	2059	2060	2061	2062	2063	2064	2065	2066	2067	2068	2069	2070	2071	2072	2073	2074	2075	2076	2077	2078	2079	2080	2081	2082	2083	2084	2085	2086	2087	2088	2089	2090	2091	2092	2093	2094	2095	2096	2097	2098	2099	2100

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	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
	592.0	100.00	0.00
5	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.00
	383.9	100.00	0.00
10	352.0	100.00	0.00
	322.8	100.00	0.00
	296.0	100.00	0.00
	271.4	100.00	0.00
	248.9	100.00	0.00
15	228.2	100.00	0.00
	209.3	100.00	0.00
	191.9	100.00	0.00
	176.0	100.00	0.00
	161.4	100.00	0.00
20	148.0	100.00	0.00
	135.7	100.00	0.00
	124.5	100.00	0.00
	114.1	100.00	0.00
	104.7	100.00	0.00
25	95.96	100.00	0.00
	88.00	100.00	0.00
	80.70	100.00	0.00
	74.00	100.00	0.00
	67.86	100.00	0.00
30	62.23	100.00	0.00
	57.06	100.00	0.00
	52.33	100.00	0.00
	47.98	100.00	0.00
	44.00	100.00	0.00
35	40.35	100.00	0.00
	37.00	100.00	0.00
	33.93	100.00	0.00
	31.11	100.00	0.16
	28.53	99.84	0.26
40	26.16	99.58	0.33
	23.99	99.25	0.42
	22.00	99.83	0.55
	20.17	99.28	0.72
	18.50	97.56	0.92
45	16.96	96.64	1.15
	15.56	95.49	1.43
	14.27	94.06	1.75
	13.08	92.31	2.10
	12.00	90.21	2.46
50	11.00	87.75	2.84
	10.09	84.91	3.18
	9.250	81.73	3.50
	8.482	78.23	3.75
	7.778	74.48	3.96
55	7.133	70.52	4.11
	6.541	66.41	4.21

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	5.998	62.20	4.29
	5.500	57.91	4.35
	5.044	53.56	4.40
	4.625	49.16	4.46
5	4.241	44.70	4.49
	3.889	40.21	4.50
	3.566	35.71	4.44
	3.270	31.27	4.30
	2.999	26.97	4.06
10	2.750	22.91	3.72
	2.522	19.19	3.34
	2.312	15.85	2.94
	2.121	12.91	2.57
	1.945	10.34	2.22
15	1.783	8.12	1.90
	1.635	6.22	1.62
	1.499	4.60	1.34
	1.375	3.26	1.08
	1.261	2.18	0.82
20	1.156	1.36	0.58
	1.060	0.78	0.39
	0.972	0.39	0.26
	0.892	0.13	0.13
	0.818	0.00	0.00
25	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
	0.530	0.00	0.00
30	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
	0.344	0.00	0.00
35	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
	0.223	0.00	0.00
40	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
	0.145	0.00	0.00
45	0.133	0.00	0.00

The final particle size achieved in this second expanded bed experiment were:

90 % of the particles had a size less than 11.91 μm ;

50 % of the particles had a size less than 6.05 μm ; and

the median value of the size of the particles was 6.05 μm

More importantly, 90.21 % of the fly ash produced with this non-expanded bed process had a size

less than 12.0 μm .

Run No. 2

Machine: HSA-30
 5 Tank: SS
 Arms: 8 L
 Diverter Discs: 3
 Tank Volume: 28.6 gallons
 Cooling Water: Yes
 10 Discharge Screen: 0.8 MM/LD
 Open Area: 1-Side 100 %
 Horse Power: 50
 RPM Setting: 400
 Power: 66 amps
 15 Original Particle Size: DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
 Desired Particle Size: 90% - 12 μm
 Process Procedure: Continuous Dry Grind
 Test Data:
 1) Material Processed: 358 lbs/15 minutes
 2) Product Temperature: 230° F
 20 3) Cooling Water Inlet temperature: 30° F / Outlet: 54° F
 4) Cooling Rate: 10 GPM

Process Rate: 1432 lbs/hour

25 The grinding medium comprises 11.5 gallons (462 lbs) of carbon steel spheres having a size of about 1/8 inch. The results of this run are set forth in Table 10, and graphically in Figure 17.

Table 10

30	MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
35	Id #1: FLY ASH 8 COARSE Distrib. Format: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 2 runs Transmission: 0.92 Laser Int: 1,008/1.002/1.004 Residuals: Disabled Above Residual: 0.00 Below Residual: 0.00	Summary Data dv = 0.0412 10% = 1.830 50% = 4.301 90% = 10.49 mv = 5.458 mn = 1.829 ma = 3.531 cs = 1.699 sd = 3.251	Id # 2: Fly ash Med Load Time: 17:23 Chan. Progression: Geom/8rt2 Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp. Spherical Particles: No Part. Refractive Index: 1.81
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<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
704.0	100.00	0.00
645.6	100.00	0.00
592.0	100.00	0.00
542.9	100.00	0.00
497.8	100.00	0.00
456.5	100.00	0.00
418.6	100.00	0.00
383.9	100.00	0.00
352.0	100.00	0.00
322.8	100.00	0.00
296.0	100.00	0.00
271.4	100.00	0.00
248.9	100.00	0.00
228.2	100.00	0.00
209.3	100.00	0.00
191.9	100.00	0.00
176.0	100.00	0.00
161.4	100.00	0.00
148.0	100.00	0.00
135.7	100.00	0.00
124.5	100.00	0.00
114.1	100.00	0.00
104.7	100.00	0.00
95.96	100.00	0.00
88.00	100.00	0.00
80.70	100.00	0.00
74.00	100.00	0.00
67.86	100.00	0.00
62.23	100.00	0.00
57.06	100.00	0.00
52.33	100.00	0.00
47.98	100.00	0.00
44.00	100.00	0.00
40.35	100.00	0.00
37.00	100.00	0.00
33.93	100.00	0.00
31.11	100.00	0.05
28.53	99.95	0.12
26.16	99.83	0.21
23.99	99.62	0.30
22.00	99.32	0.37
20.17	98.95	0.48
18.50	98.47	0.62
16.96	97.85	0.80
15.56	97.05	1.01
14.27	96.04	1.28
13.08	94.76	1.59
12.00	93.17	1.95
11.00	91.22	2.33
10.09	88.89	2.72
9.250	86.17	3.11
8.482	83.06	3.46
7.778	79.60	3.77
7.133	75.83	4.03

	6.541	71.80	4.23
	5.998	67.57	4.40
	5.500	63.17	4.53
	5.044	58.64	4.65
5	4.625	53.99	4.75
	4.241	49.24	4.82
	3.889	44.24	4.85
	3.566	39.57	4.80
	3.270	34.77	4.67
10	2.999	30.10	4.43
	2.750	25.67	4.08
	2.522	21.59	3.69
	2.312	17.90	3.26
	2.121	14.64	2.86
15	1.945	11.78	2.49
	1.783	9.29	2.15
	1.635	7.14	1.84
	1.499	5.30	1.54
	1.375	3.76	1.24
20	1.261	2.52	0.95
	1.156	1.57	0.67
	1.060	0.90	0.46
	0.972	0.44	0.29
	0.892	0.15	0.15
25	0.818	0.00	0.00
	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
30	0.530	0.00	0.00
	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
35	0.344	0.00	0.00
	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
40	0.223	0.00	0.00
	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
45	0.145	0.00	0.00
	0.133	0.00	0.00

The final particle size achieved in this second non-expanded bed were:

90 % of the particles had a size less than 10.49 μm ;

50 93.1 % of the particles had a size less than 12.0 μm ;

50 % of the particles had a size less than 4.31 μm ; and

the median value of the size of the particles was 5.45

Run No. 3

55 Machine:

HSA-30

	Tank:	SS
	Arms:	8 L
	Diverter Discs:	3
	Tank Volume:	28.6 gallons
5	Cooling Water:	Yes
	Discharge Screen:	0.8 MM/LD
	Open Area:	1-Side 100%
	Horse Power:	50
	RPM Setting:	400
10	Power:	66 amps
	Original Particle Size:	DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
	Desired Particle Size:	90% - 12 μm
	Process Procedure:	Continuous Dry Grind
15	Test Data:	1) Material Processed: 450 lbs/15 minutes
		2) Product Temperature: 206° F
		3) Cooling Water Inlet temperature: 40° F / Outlet: 50° F
		4) Cooling Rate: 10 GPM
		5) Feeder setting: 640
20	Process Rate:	1800 lbs/hour

The grinding medium comprises 11.5 gallons (462 lbs) of carbon steel spheres having a size of about 1/8 inch. The results of this run are set forth in Table 11, and graphically in Figure 18.

Table 11

25	MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
30	Id #1: FLY ASH 8 COARSE Distrib. Format: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 2 runs Transmission: 0.90 Laser Int: 1,008/1.002/1.004 Residuals: Disabled Above Residual: 0.00 Below Residual: 0.00	Summary Data dv = 0.0575 10% = 1.907 50% = 4.709 90% = 12.31 mv = 6.148 mn = 1.843 ma = 3.774 cs = 1.590 sd = 3.861	Id # 2: Fly ash Med Load Chan. Progression: Geom/8rt2 Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp. Spherical Particles: No Part. Refractive Index: 1.81
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	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
	592.0	100.00	0.00
5	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.00
	383.9	100.00	0.00
10	352.0	100.00	0.00
	322.8	100.00	0.00
	296.0	100.00	0.00
	271.4	100.00	0.00
	248.9	100.00	0.00
15	228.2	100.00	0.00
	209.3	100.00	0.00
	191.9	100.00	0.00
	176.0	100.00	0.00
	161.4	100.00	0.00
20	148.0	100.00	0.00
	135.7	100.00	0.00
	124.5	100.00	0.00
	114.1	100.00	0.00
	104.7	100.00	0.00
25	95.96	100.00	0.00
	88.00	100.00	0.00
	80.70	100.00	0.00
	74.00	100.00	0.00
	67.86	100.00	0.00
30	62.23	100.00	0.00
	57.06	100.00	0.00
	52.33	100.00	0.00
	47.98	100.00	0.00
	44.00	100.00	0.00
35	40.35	100.00	0.00
	37.00	100.00	0.05
	33.93	99.95	0.10
	31.11	99.85	0.19
	28.53	99.66	0.26
40	26.16	99.40	0.34
	23.99	99.06	0.46
	22.00	98.60	0.61
	20.17	97.99	0.79
	18.50	97.20	1.02
45	16.96	96.18	1.28
	15.56	94.90	1.56
	14.27	93.34	1.84
	13.08	91.50	2.14
	12.00	89.36	2.44
50	11.00	86.92	2.73
	10.09	84.19	3.03
	9.250	81.16	3.31
	8.482	77.85	3.57
	7.778	74.28	3.81
55	7.133	70.47	4.02

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	6.541	66.45	4.18
	5.998	62.27	4.31
	5.500	57.96	4.40
	5.044	53.56	4.48
5	4.625	49.08	4.52
	4.241	44.56	4.54
	3.889	40.02	4.51
	3.566	35.51	4.41
	3.270	31.10	4.23
10	2.999	26.87	3.98
	2.750	22.89	3.65
	2.522	19.24	3.28
	2.312	15.96	2.89
	2.121	13.07	2.54
15	1.945	10.53	2.21
	1.783	8.32	1.91
	1.635	6.41	1.64
	1.499	4.77	1.37
	1.375	3.40	1.11
20	1.261	2.29	0.85
	1.156	1.44	0.61
	1.060	0.83	0.42
	0.972	0.41	0.27
	0.892	0.14	0.14
25	0.818	0.00	0.00
	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
30	0.530	0.00	0.00
	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
35	0.344	0.00	0.00
	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
40	0.223	0.00	0.00
	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
45	0.145	0.00	0.00
	0.133	0.00	0.00

The final particle size achieved in this third non-expanded bed were:

- 50 90 % of the particles had a size less than 12.31 μm ;
 89.3 % of the particles had a size less than 12.0 μm ;
 50 % of the particles had a size less than 4.70 μm ; and
 the median value of the size of the particles was 6.14

55 Run No.4

	Machine:	HSA-30
	Tank:	SS
	Arms:	8 L
	Diverter Discs:	3
5	Tank Volume:	28.6 gallons
	Cooling Water:	Yes
	Discharge Screen:	0.8 MM/LD
	Open Area:	1-Side 100 %
	Horse Power:	50
10	RPM Setting:	400
	Power:	66 amps
	Original Particle Size:	DCF 50% 16.3 μ m / MV 30.08 μ m / 90 % 74.44 μ m
	Desired Particle Size:	90% - 12 μ m
	Process Procedure:	Continuous Dry Grind
15	Test Data:	1) Material Processed: 500 lbs/15 minutes 2) Product Temperature: 200° F 3) Cooling Water Inlet temperature: 40° F / Outlet: 50° F 4) Cooling Rate: 10 GPM 5) Feeder setting: 720
20	Process Rate:	2000 lbs/hour

The grinding medium comprises 11.5 gallons (462 lbs) of carbon steel spheres having a size of about 1/8 inch. The results of this run are set forth in Table 12 below, and graphically in figure 19.

Table 12

MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
30	Id #1: FLY ASH 8 COARSE Distrib. Format: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 2 runs Transmission: 0.93 Laser Int: 1,008/1.002/1.004 Residuals: Disabled Above Residual: 0.00 Below Residual: 0.00	Summary Data dv = 0.0393 10% = 1.969 50% = 4.912 90% = 12.55 mv = 6.301 mn = 1.873 ma = 3.899 cs = 1.539 sd = 3.965
35		Id # 2: Fly ash Med Load Chan. Progression: Geom/8rt2 Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp. Spherical Particles: No Part. Refractive Index: 1.81

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
5	592.0	100.00	0.00
	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.00
10	383.9	100.00	0.00
	352.0	100.00	0.00
	322.8	100.00	0.00
	296.0	100.00	0.00
	271.4	100.00	0.00
15	248.9	100.00	0.00
	228.2	100.00	0.00
	209.3	100.00	0.00
	191.9	100.00	0.00
	176.0	100.00	0.00
20	161.4	100.00	0.00
	148.0	100.00	0.00
	135.7	100.00	0.00
	124.5	100.00	0.00
	114.1	100.00	0.00
25	104.7	100.00	0.00
	95.96	100.00	0.00
	88.00	100.00	0.00
	80.70	100.00	0.00
	74.00	100.00	0.00
30	67.86	100.00	0.00
	62.23	100.00	0.00
	57.06	100.00	0.00
	52.33	100.00	0.00
	47.98	100.00	0.00
35	44.00	100.00	0.00
	40.35	100.00	0.00
	37.00	100.00	0.00
	33.93	100.00	0.00
	31.11	100.00	0.17
40	28.53	99.83	0.28
	26.16	99.55	0.35
	23.99	99.20	0.48
	22.00	98.72	0.65
	20.17	98.07	0.86
45	18.50	97.21	1.11
	16.96	96.10	1.37
	15.56	94.73	1.67
	14.27	93.06	1.98
	13.08	91.08	2.30
50	12.00	88.78	2.63
	11.00	86.15	2.95
	10.09	83.20	3.26
	9.250	79.94	3.55
	8.482	76.39	3.79
55	7.778	72.60	4.00
	7.133	68.60	4.17

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	6.541	64.43	4.28
	5.998	60.15	4.37
	5.500	55.78	4.41
	5.044	51.37	4.45
5	4.625	46.92	4.46
	4.241	42.46	4.44
	3.889	38.02	4.39
	3.566	33.63	4.28
	3.270	29.35	4.09
10	2.999	25.26	3.83
	2.750	21.43	3.49
	2.522	17.94	3.12
	2.312	14.82	2.75
	2.121	12.07	2.39
15	1.945	9.68	2.07
	1.783	7.61	1.78
	1.635	5.83	1.51
	1.499	4.32	1.25
	1.375	3.07	1.00
20	1.261	2.07	0.77
	1.156	1.30	0.55
	1.060	0.75	0.37
	0.972	0.38	0.25
	0.892	0.13	0.13
25	0.818	0.00	0.00
	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
30	0.530	0.00	0.00
	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
35	0.344	0.00	0.00
	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
40	0.223	0.00	0.00
	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
45	0.145	0.00	0.00
	0.133	0.00	0.00

The final particle size achieved in this fourth non-expanded bed were:

90 % of the particles had a size less than 12.55 μm ;

50 88.7 % of the particles had a size less than 12.0 μm ;

50 % of the particles had a size less than 4.91 μm ; and

the median value of the size of the particles was 6.30 μm .

Run No. 5

Machine: HSA-30
 Tank: SS
 Arms: 8 L
 5 Diverter Discs: 3
 Tank Volume: 28.6 gallons
 Cooling Water: Yes
 Discharge Screen: 0.8 MM/LD
 Open Area: 1-Side 100%
 10 Horse Power: 50
 RPM Setting: 400
 Power: 65 amps
 Original Particle Size: DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
 Desired Particle Size: 90% - 12 μm
 15 Process Procedure: Continuous Dry Grind
 Test Data:
 1) Material Processed: 550 lbs/15 minutes
 2) Product Temperature: 195° F
 3) Cooling Water Inlet temperature: 42° F / Outlet: 58° F
 4) Cooling Rate: 10 GPM
 20 5) Feeder setting: 800
 Process Rate: 2200 lbs/hour

The grinding medium comprises 11.5 gallons (462 lbs) of carbon steel spheres having a size of about 1/8 inch. The results of this run are set forth in Table 13 below, and graphically in figure

25 20.

Table 13

MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
30	Id #1: FLY ASH 8 COARSE Distrib. Format: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 2 runs Transmission: 0.93 Laser Int: 1.008/1.002/1.004 Residuals: Disabled Above Residual: 0.00 Below Residual: 0.00	Summary Data dv = 0.0553 10% = 1.985 50% = 5.096 90% = 13.24 mv = 6.592 mn = 1.856 ma = 3.985 cs = 1.506 sd = 4.220
35		Id # 2: Fly ash Med Load Chan. Progression: Geom/8rt2 Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp. Spherical Particles: No Part. Refractive Index: 1.81
40		

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
	592.0	100.00	0.00
5	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.00
	383.9	100.00	0.00
10	352.0	100.00	0.00
	322.8	100.00	0.00
	296.0	100.00	0.00
	271.4	100.00	0.00
	248.9	100.00	0.00
15	228.2	100.00	0.00
	209.3	100.00	0.00
	191.9	100.00	0.00
	176.0	100.00	0.00
	161.4	100.00	0.00
20	148.0	100.00	0.00
	135.7	100.00	0.00
	124.5	100.00	0.00
	114.1	100.00	0.00
	104.7	100.00	0.00
25	95.96	100.00	0.00
	88.00	100.00	0.00
	80.70	100.00	0.00
	74.00	100.00	0.00
	67.86	100.00	0.00
30	62.23	100.00	0.00
	57.06	100.00	0.00
	52.33	100.00	0.00
	47.98	100.00	0.00
	44.00	100.00	0.00
35	40.35	100.00	0.00
	37.00	100.00	0.05
	33.93	99.95	0.12
	31.11	99.83	0.22
	28.53	99.61	0.32
40	26.16	99.29	0.43
	23.99	98.86	0.58
	22.00	98.28	0.77
	20.17	97.51	1.00
	18.50	96.51	1.26
45	16.96	95.25	1.55
	15.56	93.70	1.85
	14.27	91.85	2.16
	13.08	89.69	2.48
	12.00	87.21	2.79
50	11.00	84.42	3.09
	10.09	81.33	3.36
	9.250	77.97	3.62
	8.482	74.35	3.83
	7.778	70.52	4.01
55	7.133	66.51	4.14

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	6.541	62.37	4.24
	5.998	58.13	4.30
	5.500	53.83	4.34
	5.044	49.49	4.35
5	4.625	45.14	4.34
	4.241	40.80	4.30
	3.889	36.50	4.24
	3.566	32.26	4.10
	3.270	28.16	3.90
10	2.999	24.26	3.64
	2.750	20.62	3.30
	2.522	17.32	2.96
	2.312	14.36	2.60
	2.121	11.76	2.27
15	1.945	9.49	1.97
	1.783	7.52	1.71
	1.635	5.81	1.46
	1.499	4.35	1.23
	1.375	3.12	1.00
20	1.261	2.12	0.77
	1.156	1.35	0.56
	1.060	0.79	0.39
	0.972	0.40	0.26
	0.892	0.14	0.14
25	0.818	0.00	0.00
	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
30	0.530	0.00	0.00
	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
35	0.344	0.00	0.00
	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
40	0.223	0.00	0.00
	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
45	0.145	0.00	0.00
	0.133	0.00	0.00

The final particle size achieved in this fifth non-expanded bed were:

90 % of the particles had a size less than 13.24 μm ;

50 87.21 % of the particles had a size less than 12.0 μm ;

50 % of the particles had a size less than 5.09 μm ; and

the median value of the size of the particles was 6.30 μm .

Run No. 6

Machine: HSA-30
 Tank: SS
 Arms: 8 L
 5 Diverter Discs: 3
 Tank Volume: 28.6 gallons
 Cooling Water: Yes
 Discharge Screen: 0.8 MM/LD
 Open Area: 1-Side 100%
 10 Horse Power: 50
 RPM Setting: 400
 Power: 66 amps
 Original Particle Size: DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
 Desired Particle Size: 90% - 12 μm
 15 Process Procedure: Continuous Dry Grind
 Test Data:
 1) Material Processed: 570 lbs/15 minutes
 2) Product Temperature: 195° F
 3) Cooling Water Inlet temperature: 30° F / Outlet: 48° F
 4) Cooling Rate: 10 GPM
 20 5) Feeder setting: 800
 Process Rate: 2280 lbs/hour

The grinding medium comprises 11.5 gallons (462 lbs) of carbon steel spheres having a size of about 1/8 inch. The results of this run are set forth in Table 14 below, and graphically in figure

25 21.

Table 14

MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
30	Id #1: FLY ASH 8 COARSE	Summary Data
	Distrib. Format: Volume	dv = 0.0564
35	Filter: On	10% = 1.945
	Run Time: 60 seconds	50% = 5.012
40	Run Number: Avg of 2 runs	90% = 13.59
	Transmission: 0.90	mv = 6.688
	Laser Int: 1.008/1.002/1.004	mn = 1.830
	Residuals: Disabled	ma = 3.929
	Above Residual: 0.000	cs = 1.527
	Below Residual: 0.000	sd = 4.309
	Id # 2: Fly Ash Med Load	
	Time: 13:00	
	Chan. Progression: Geom/8rt2	
	Upper Channel Edge: 704.0	
	Lower Channel Edge: 0.122	
	Number of Channels: 100	
	Fluid Refractive Index: 1.33	
	Particle Transparency: Transp.	
	Spherical Particles: No	
	Part. Refractive Index: 1.81	

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
5	592.0	100.00	0.00
	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.00
10	383.9	100.00	0.00
	352.0	100.00	0.00
	322.8	100.00	0.00
	296.0	100.00	0.00
	271.4	100.00	0.00
15	248.9	100.00	0.00
	228.2	100.00	0.00
	209.3	100.00	0.00
	191.9	100.00	0.00
	176.0	100.00	0.00
20	161.4	100.00	0.00
	148.0	100.00	0.00
	135.7	100.00	0.00
	124.5	100.00	0.00
	114.1	100.00	0.00
25	104.7	100.00	0.00
	95.96	100.00	0.00
	88.00	100.00	0.00
	80.70	100.00	0.00
	74.00	100.00	0.00
30	67.86	100.00	0.00
	62.23	100.00	0.00
	57.06	100.00	0.00
	52.33	100.00	0.00
	47.98	100.00	0.00
35	44.00	100.00	0.05
	40.35	99.95	0.10
	37.00	99.85	0.18
	33.93	99.67	0.24
	31.11	99.43	0.29
40	28.53	99.14	0.37
	26.16	98.77	0.48
	23.99	98.29	0.62
	22.00	97.67	0.80
	20.17	96.87	1.03
45	18.50	95.84	1.29
	16.96	94.55	1.56
	15.56	92.99	1.84
	14.27	91.15	2.12
	13.08	89.03	2.38
50	12.00	86.65	2.65
	11.00	84.00	2.91
	10.09	81.09	3.16
	9.250	77.93	3.42
	8.482	74.51	3.65
55	7.778	70.86	3.86
	7.133	67.00	4.02

	6.541	62.98	4.15
	5.998	58.83	4.23
	5.500	54.60	4.28
	5.044	50.32	4.31
5	4.625	46.01	4.32
	4.241	41.69	4.29
	3.889	37.40	4.24
	3.566	33.16	4.13
	3.270	29.03	3.94
10	2.999	25.09	3.69
	2.750	21.40	3.37
	2.522	18.03	3.02
	2.312	15.01	2.67
	2.121	12.34	2.34
15	1.945	10.00	2.05
	1.783	7.95	1.78
	1.635	6.17	1.54
	1.499	4.63	1.29
	1.375	3.34	1.06
20	1.261	2.28	0.83
	1.156	1.45	0.60
	1.060	0.85	0.42
	0.972	0.43	0.28
	0.892	0.15	0.15
25	0.818	0.00	0.00
	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
30	0.530	0.00	0.00
	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
35	0.344	0.00	0.00
	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
40	0.223	0.00	0.00
	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
45	0.145	0.00	0.00
	0.133	0.00	0.00

The final particle size achieved in this sixth non-expanded bed were:

90 % of the particles had a size less than 13.59 μm ;

50 85.65 % of the particles had a size less than 12.0 μm ;

50 % of the particles had a size less than 5.01 μm ; and

the median size of a particle produced in this run was 6.68 μm .

Run No. 7

Machine: HSA-30
 Tank: SS
 Arms: 8 L
 5 Diverter Discs: 3
 Tank Volume: 28.6 gallons
 Cooling Water: Yes
 Discharge Screen: 0.8 MM/LD
 Open Area: 1-Side 100 %
 10 Horse Power: 50
 RPM Setting: 400
 Power: 66 amps
 Original Particle Size: DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
 Desired Particle Size: 90% - 12 μm
 15 Process Procedure: Continuous Dry Grind
 Test Data:
 1) Material Processed: 675 lbs/15 minutes
 2) Product Temperature: 195° F
 3) Cooling Water Inlet temperature: 30° F / Outlet: 48° F
 4) Cooling Rate: 10 GPM
 20 Process Rate: 2700 lbs/hour

The grinding medium comprises 11.5 gallons (462 lbs) of carbon steel spheres having a size of about 1/8 inch. The results of this run are set forth in Table 15 below, and graphically in figure

25 22.

Table 15

MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
Id #1: FLY ASH 8 COARSE Distrib. Format: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 2 runs Transmission: 0.90 Laser Int: 1,008/1.002/1.004 Residuals: Disabled Above Residual: 0.000 Below Residual: 0.000	Summary Data dv = 0.0618 10% = 1.837 50% = 4.564 90% = 10.97 mv = 5.698 mn = 1.805 ma = 3.629 cs = 1.653 sd = 3.459	Id # 2: Fly Ash Med Load Time: 13:00 Chan. Progression: Geom/8rt2 Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp. Spherical Particles: No Part. Refractive Index: 1.81

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
5	592.0	100.00	0.00
	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.00
10	383.9	100.00	0.00
	352.0	100.00	0.00
	322.8	100.00	0.00
	296.0	100.00	0.00
	271.4	100.00	0.00
15	248.9	100.00	0.00
	228.2	100.00	0.00
	209.3	100.00	0.00
	191.9	100.00	0.00
	176.0	100.00	0.00
20	161.4	100.00	0.00
	148.0	100.00	0.00
	135.7	100.00	0.00
	124.5	100.00	0.00
	114.1	100.00	0.00
25	104.7	100.00	0.00
	95.96	100.00	0.00
	88.00	100.00	0.00
	80.70	100.00	0.00
	74.00	100.00	0.00
30	67.86	100.00	0.00
	62.23	100.00	0.00
	57.06	100.00	0.00
	52.33	100.00	0.00
	47.98	100.00	0.00
35	44.00	100.00	0.00
	40.35	100.00	0.00
	37.00	100.00	0.00
	33.93	100.00	0.00
	31.11	100.00	0.05
40	28.53	99.95	0.12
	26.16	99.83	0.21
	23.99	99.62	0.31
	22.00	99.31	0.40
	20.17	98.91	0.53
45	18.50	98.38	0.71
	16.96	97.67	0.92
	15.56	96.75	1.18
	14.27	95.57	1.48
	13.08	94.09	1.82
50	12.00	92.27	2.20
	11.00	90.07	2.61
	10.09	87.46	3.02
	9.250	84.44	3.43
	8.482	81.01	3.80
55	7.778	77.21	4.10
	7.133	73.11	4.33

	6.541	68.78	4.47
	5.998	64.31	4.53
	5.500	59.78	4.53
	5.044	55.25	4.54
5	4.625	50.71	4.54
	4.241	46.17	4.53
	3.889	41.64	4.49
	3.566	37.15	4.40
	3.270	32.75	4.26
10	2.999	28.49	4.03
	2.750	24.46	3.74
	2.522	20.72	3.40
	2.312	17.32	3.04
	2.121	14.28	2.69
15	1.945	11.59	2.37
	1.783	9.22	2.07
	1.635	7.15	1.79
	1.499	5.36	1.52
	1.375	3.84	1.24
20	1.261	2.60	0.96
	1.156	1.64	0.69
	1.060	0.95	0.48
	0.972	0.47	0.31
	0.892	0.16	0.16
25	0.818	0.00	0.00
	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
30	0.530	0.00	0.00
	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
35	0.344	0.00	0.00
	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
40	0.223	0.00	0.00
	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
45	0.145	0.00	0.00
	0.133	0.00	0.00

The final particle size achieved in this seventh non-expanded bed were:

- 50 90 % of the particles had a size less than 10.97 μm ;
 92.2 % of the particles had a size less than 12.0 μm ;
 50 % of the particles had a size less than 4.56 μm ; and
 the median size of a particle produced in this run was 5.69 μm .

55 Run No. 8

	Machine:	HSA-30
	Tank:	SS
	Arms:	8 L
	Diverter Discs:	3
5	Tank Volume:	28.6 gallons
	Cooling Water:	Yes
	Discharge Screen:	0.8 MM/LD
	Open Area:	1-Side 100%
	Horse Power:	50
10	RPM Setting:	400
	Power:	66 amps
	Original Particle Size:	DCF 50% 16.3 μm / MV 30.08 μm / 90 % 74.44 μm
	Desired Particle Size:	90% - 12 μm
	Process Procedure:	Continuous Dry Grind
15	Test Data:	1) Material Processed: 675 lbs/15 minutes 2) Product Temperature: 195° F 3) Cooling Water Inlet temperature: 30° F / Outlet: 48° F 4) Cooling Rate: 10 GPM
20	Process Rate:	2700 lbs/hour

The grinding medium comprises 11.5 gallons (462 lbs) of carbon steel spheres having a size of about 1/8 inch. The results of this run are set forth in Table 16 below, and graphically in figure 23.

Table 16

MICROTRAC X100 PARTICLE ANALYZER PERCENT PASSING DATA ASVR PARAMETERS		
30	Id #1: FLY ASH 8 COARSE Distrib. Format: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 2 runs Transmission: 0.90 Laser Int: 1,008/1.002/1.004 Residuals: Disabled Above Residual: 0.000 Below Residual: 0.000	Summary Data dv = 0.0663 10% = 1.967 50% = 5.156 90% = 13.50 mv = 6.706 mn = 1.832 ma = 3.988 cs = 1.504 sd = 4.328
35		Id # 2: Fly ash Med Load Time: 13:00 Chan. Progression: Geom/8rt2 Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp. Spherical Particles: No Part. Refractive Index: 1.81

	<u>Ch Top</u>	<u>% Pass</u>	<u>% Change</u>
	704.0	100.00	0.00
	645.6	100.00	0.00
5	592.0	100.00	0.00
	542.9	100.00	0.00
	497.8	100.00	0.00
	456.5	100.00	0.00
	418.6	100.00	0.00
10	383.9	100.00	0.00
	352.0	100.00	0.00
	322.8	100.00	0.00
	296.0	100.00	0.00
	271.4	100.00	0.00
15	248.9	100.00	0.00
	228.2	100.00	0.00
	209.3	100.00	0.00
	191.9	100.00	0.00
	176.0	100.00	0.00
20	161.4	100.00	0.00
	148.0	100.00	0.00
	135.7	100.00	0.00
	124.5	100.00	0.00
	114.1	100.00	0.00
25	104.7	100.00	0.00
	95.96	100.00	0.00
	88.00	100.00	0.00
	80.70	100.00	0.00
	74.00	100.00	0.00
30	67.86	100.00	0.00
	62.23	100.00	0.00
	57.06	100.00	0.00
	52.33	100.00	0.00
	47.98	100.00	0.00
35	44.00	100.00	0.00
	40.35	100.00	0.00
	37.00	100.00	0.14
	33.93	99.86	0.21
	31.11	99.65	0.26
40	28.53	99.39	0.33
	26.16	99.06	0.45
	23.99	98.61	0.60
	22.00	98.01	0.79
	20.17	97.22	1.03
45	18.50	96.19	1.30
	16.96	94.89	1.60
	15.56	93.29	1.91
	14.27	91.38	2.22
	13.08	89.16	2.53
50	12.00	86.63	2.84
	11.00	83.79	3.13
	10.09	80.66	3.40
	9.250	77.26	3.65
	8.482	73.61	3.85
55	7.778	69.76	4.02
	7.133	65.74	4.14

	6.541	61.60	4.19
	5.998	57.41	4.23
	5.500	53.18	4.25
	5.044	48.93	4.24
5	4.625	44.69	4.22
	4.241	40.47	4.17
	3.889	36.30	4.12
	3.566	32.18	4.00
10	3.270	28.18	3.82
	2.999	24.36	3.58
	2.750	20.78	3.27
	2.522	17.51	2.93
	2.312	14.58	2.59
	2.121	11.99	2.27
15	1.945	9.72	1.99
	1.783	7.73	1.72
	1.635	6.01	1.49
	1.499	4.52	1.26
	1.375	3.26	1.03
20	1.261	2.23	.080
	1.156	1.43	0.59
	1.060	0.84	0.41
	0.972	0.43	0.28
	0.892	0.15	0.15
25	0.818	0.00	0.00
	0.750	0.00	0.00
	0.688	0.00	0.00
	0.630	0.00	0.00
	0.578	0.00	0.00
30	0.530	0.00	0.00
	0.486	0.00	0.00
	0.446	0.00	0.00
	0.409	0.00	0.00
	0.375	0.00	0.00
35	0.344	0.00	0.00
	0.315	0.00	0.00
	0.289	0.00	0.00
	0.265	0.00	0.00
	0.243	0.00	0.00
40	0.223	0.00	0.00
	0.204	0.00	0.00
	0.187	0.00	0.00
	0.172	0.00	0.00
	0.158	0.00	0.00
45	0.145	0.00	0.00
	0.133	0.00	0.00

The final particle size achieved in this eighth non-expanded bed were:

90 % of the particles had a size less than 13.50 μm ;

50 86.6 % of the particles had a size less than 12.0 μm ;

50 % of the particles had a size less than 5.15 μm ; and

the median size of a particle produced in this run was 6.70 μm .

Results

Table 17

Grinding Results at 600 RPM

Grinding Medium volume	Amperes	Feed Setting	Product. Rate (lbs/hr)	% bed voids	50 % Particle Size	% less than 12 μ	Product Discharge Temp. (F)	Product Discharge Opening
5.7	65	440	1050	-----	5.2 μ	81	260	100 % one side
5.7	65	555	1440	-----	6.7 μ	72	230	100 % one side
5.7	65	640	1800	-----	7.7 μ	66	210	100 % one side

Operation of the mill at 600 RPM produces an expanded bed where grinding effectiveness of the bed decreases with an increase in feed setting. At 600 RPM, it appears that the smallest feed setting to the bed volume produces the finer particle size distribution, but at lower production rates when compared to using the higher feed settings (compare curves representing % volume difference in Figures 13, 14, and 15). A comparison of these figures show that the particle size distributions become broader and coarser with increasing feed setting. At the lowest feed setting, the distribution of particle sizes show that 10% of the particles are smaller than 1.3 μ m, and 90% of the particles are less than 17.7 μ m (see Figure 13). At the 555 feed setting, 10% of the particles are smaller than 1.7 μ m, and 90% of the particles are less than 29.8 μ m (see Figure 14). At the 640 feed setting, 10% of the particles are also below 1.7 μ m, but 90% of the particles are less than 35 μ m. In the expanded bed tests, the highest feed setting introduced sufficient fly ash to exceed the bed void volume. The reduction in bed void volume, due to increased feed settings of fly ash, leads to a coarser product.

However, reduction of the mill speed from 600 RPM to 400 RPM, while maintaining the current constant at 65 amps, allowed the volume of the grinding media to be increased from 5.7 gallons to 11.5 gallons. In addition, at 400 RPM, the volume of the bed does not expand.

Under the conditions described in Table 18 below, the particle size distribution within the product does not appear to be influenced by the feed rate over the range of feed settings from 440 up to 990. This may be seen by comparison of the % volume cumulative curves in Figures 16 through 23. In the first 3 runs the grinding process was operated in the expanded bed mode. The feed rate was increased from 440 to 640, which increased the rate of fly ash processed. As a result, the

volume of fly ash to the void volume increased, and efficiency of the grinding decreased, which can be seen in Table 17 above.

In contrast, operation of the bed in a non-expanded mode as the feed setting increased employed in these tests indicates that the contact time between the media and the fly ash is unaffected by the increase in feed setting or influenced by a reduction in the product discharging opening. Furthermore, operation at the maximum feed setting of 990, and employing a product discharging opening of 25% of one side still does not provide sufficient quantities of fly ash within the bed to reduce the contact time between the media and the fly ash to produce a coarser product as was observed with the expanded bed tests set forth above. This is apparently due to the grinding condition where the fly ash in the grinding bed does not exceed the void volume.

In the case of the tests using the expanded bed and increased feed settings, the contact time between the media and the fly ash becomes shortened with an increase in feed setting. A comparison of the % volume different curves in Figures 13, 14 and 15 show that the particle size distributions range become broader and coarser as the feed settings are increased.

Also, it should be noted that in Table 18, as the feed settings are increased, the bed temperatures decrease from 260° F to 195° F. Apparently, the greater rate of the fly ash process in the non-expanded mode provides a heat sink and transfer of grinding heat to the wall of the tank.

Table 18

Grinding Results at 400 RPM

bed med wt (lbs)	bed med. Vol. (Gal)	amps	feed setting	Prod Rate lbs/hr	%bed voids	bed prod. Vol.	50 % particle size(μ)	% of particle s < 12 μ	Prod. Disch. Temp.	Prod. Dis. Open.
462	11.5	65	440	1050	49	2.4	4.7	90.2	260	100
462	11.5	65	550	1440	49	2.4	4.3	93.2	230	100
462	11.5	65	640	1800	49	2.4	4.7	89.4	206	100
462	11.5	65	720	2000	49	2.4	4.9	88.9	200	100
462	11.5	65	800	2280	49	2.4	5.0	87.2	195	100
462	11.5	65	800	2280	37	3.0	5.0	86.7	195	50
462	11.5	65	990	2700	37	3.0	4.6	92.3	195	50
462	11.5	65	990	2700	16	4.0	5.2	86.6	195	25

Significance of the Results

Using a non-expanded bed of the invention, as much as 2700 lbs/ hour of coarse fly ash can be continuously ground to produce a fly ash product with acceptable particles size distributions for use in concrete or mortar, wherein 87 % or greater of the particles have a size of 12 μm or less. The present invention is not to be limited in scope by the specific embodiments describe herein.

- 5 Indeed, various modifications of the invention in addition to those described herein will become apparent to those skilled in the art from the foregoing description and the accompanying figures. Such modifications are intended to fall within the scope of the appended claims.

- 10 Various publications are cited herein, the disclosures of which are incorporated by reference in their entireties.

561210: 064E.000

WHAT IS CLAIMED IS:

1. Fly ash characterized by
 - a) substantially uniform spherical shape;
 - 5 b) greater than about 90 % of the particles have a diameter of less than 11 μm , greater than about 60 % of the particles have a diameter of less than 5.5 μm , and greater than about 15 % of the particles have a diameter of less than 1.375 μm ;
 - c) a median particle diameter of less than about 4.0 μm ; and
 - d) a range of particle diameters of from about 0.1 μm to about 70 μm .
- 10 2. The fly ash of claim 1, wherein greater than about 93 % of the particles have a diameter of less than 11 μm , greater than about 70 % of the particles have a diameter of less than 5.5 μm , and greater than about 18 % of the particles have a diameter of less than 1.375 μm .
- 15 3. The fly ash of claim 1, wherein the median particle diameter is less than about 3.0 μm .
4. The fly ash of claim 1, wherein the range of particle diameters is from about 0.9 μm to about 62 μm .
- 20 5. The fly ash of claim 1, wherein
 - a) greater than about 93 % of the particles have a diameter of less than 11 μm , greater than about 70 % of the particles have a diameter of less than 5.5 μm , and greater than about 18 % of the particles have a diameter of less than 1.375 μm ;
 - 25 b) the median particle diameter is less than about 3.0 μm ; and
 - c) the range of particle diameters is from about 0.9 μm to about 62 μm .
6. The fly ash of claim 5, which is prepared by grinding unfractionated fly ash.
- 30 7. A concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10 % to about 50 % by weight the fly ash of claim 1 and about 50 % to about 90 % by weight cement.

8. A concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the fly ash of claim 5 and about 50% to about 90% by weight cement.
9. The concrete of claim 7 further comprising silica fume.
10. The concrete of claim 7 further comprising glass fibers.
11. A mortar comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the fly ash of claim 1 and about 50% to about 90% by weight cement.
12. A mortar comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the fly ash of claim 5 and about 50% to about 90% by weight cement.
13. The mortar of claim 12 further comprising silica fume.
14. The mortar of claim 12 further comprising glass fibers.
15. Fly ash prepared by processing fly ash so as to shift the size distribution to have the following characteristics:
 - a) substantially uniform spherical shape; greater than about 90% of the particles have a diameter of less than 11 μm , greater than about 60% of the particles have a diameter of less than 5.5 μm , and greater than about 15% of the particles have a diameter of less than 1.375 μm ;
 - b) a median particle diameter of less than about 4.0 μm ; and
 - c) a range of particle diameters of from about 0.1 μm to about 70 μm .
16. The fly ash of claim 15 wherein the processing comprises grinding with a fluidized bed grinding process using a ratio of one part unfractionated fly ash with four parts grinding

media (by volume).

17. The fly ash of claim 16 wherein the grinding media is zirconium silicate.
- 5 18. The fly ash of claim 16 wherein the grinding media is carbon steel.
19. A concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials
10 comprise from about 10% to about 50% by weight the fly ash of claim 15 and about 50% to about 90% by weight cement.
20. The concrete of Claim 19, further comprising silica fume.
- 15 21. The concrete of Claim 19, further comprising glass fibers.
22. A concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials
20 comprise from about 10% to about 50% by weight the fly ash of claim 17 and about 50% to about 90% by weight cement.
23. The concrete of claim 22 further comprising silica fume.
- 25 24. The concrete of claim 22 further comprising glass fibers.
25. A mortar comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the
30 fly ash of claim 15 and about 50% to about 90% by weight cement.
26. A mortar comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the
35 fly ash of claim 17 and about 50% to about 90% by weight cement.

27. The mortar of claim 26 further comprising silica fume.
28. The mortar of claim 26 further comprising glass fibers.
- 5 29. A method for preparing fly ash comprising processing fly ash so as to shift the size distribution to have the following characteristics:
 - a) substantially uniform spherical shape;
 - b) greater than about 90 % of the particles have a diameter of less than 11 μm , greater than about 60 % of the particles have a diameter of less than 5.5 μm , and greater than about 10 15 % of the particles have a diameter of less than 1.375 μm ;
 - c) a median particle diameter of less than about 4.0 μm ; and
 - d) a range of particle diameters of from about 0.1 μm to about 70 μm .
30. The method according to claim 29 wherein the processing comprises grinding with a 15 fluidized bed grinding process using a ratio of one part unfractionated fly ash with four parts grinding media (by volume).
31. The method according to claim 30, wherein the grinding media is zirconium silicate.
- 20 32. The method according to claim 30 wherein the grinding media is carbon steel.
33. The method according to claim 29 wherein the fly ash is dry bottom boiler fly ash.
34. The method according to claim 29 wherein the fly ash is wet bottom boiler fly ash.
- 25 35. Fly ash prepared by processing fly ash so as to shift the size distribution to have the following characteristics:
 - a) substantially uniform spherical shape;
 - b) greater than about 90 % of the particles have a diameter of less than 12 μm , greater than 30 about 50 % of the particles have a diameter of less than 5 μm , and greater than about 15 % of the particles have a diameter of less than 2.3 μm ;
 - c) a median particle diameter of less than about 6.0 μm ; and
 - d) a range of particle diameters of from about 0.78 μm to about 30 μm .
- 35 36. The fly ash of claim 35, wherein the processing comprises grinding the fly ash with a

grinding medium in a non-expanded bed, and the volume of fly ash is less than the void volume of the grinding medium.

37. The fly ash of Claim 36, wherein the ratio of fly ash to grinding medium is about 1 part fly ash to about 4 parts grinding medium, by volume.
38. The fly ash of Claim 36, wherein the ratio of fly ash to grinding medium is about 1 part fly ash to about 18 parts grinding medium, by weight.
39. The fly ash of claim 36 wherein the grinding media comprises carbon steel or stainless steel.
40. A concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the fly ash of claim 35 and about 50% to about 90% by weight cement.
41. The concrete of Claim 40, further comprising silica fume, glass fibers, or a combination thereof.
42. A concrete comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, about 1 to about 5 parts by weight coarse aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the fly ash of claim 36 and about 50% to about 90% by weight cement.
43. The concrete of Claim 42, wherein the ratio of fly ash to grinding medium is about 1 part fly ash to about 4 parts grinding medium, by volume.
44. The concrete of Claim 42, wherein the ratio of fly ash to grinding medium is about 1 part fly ash to about 18 parts grinding medium, by weight.
45. The concrete of Claim 42, wherein the grinding medium comprises carbon steel.

46. The concrete of Claim 42, further comprising silica fume, glass fibers, or a combination thereof.
47. A mortar comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the fly ash of claim 35 and about 50% to about 90% by weight cement.
48. A mortar comprising about 1 part by weight cementitious materials, about 1 to about 3 parts by weight fine aggregate, and about 0.35 to about 0.6 parts by weight water, wherein the cementitious materials comprise from about 10% to about 50% by weight the fly ash of claim 36 and about 50% to about 90% by weight cement.
49. The mortar of Claim 48, wherein the ratio of fly ash to grinding medium is about 1 part fly ash to about 4 parts grinding medium by volume.
50. The mortar of Claim 48, wherein the ratio of fly ash to grinding medium is about 1 part fly ash to about 18 parts grinding medium, by weight.
51. The mortar of Claim 48, wherein the grinding medium is carbon steel.
52. The mortar of Claim 48, wherein the grinding medium is stainless steel.
53. The mortar of Claim 48, further comprising silica fume, glass fibers, or a combination thereof.
54. A method for preparing fly ash comprising processing fly ash so as to shift the size distribution to have the following characteristics:
 - a) substantially uniform spherical shape;
 - b) greater than about 90% of the particles have a diameter of less than 12 μm , greater than about 50% of the particles have a diameter of less than 5 μm , and greater than about 15% of the particles have a diameter of less than 2.3 μm ;
 - c) a median particle diameter of less than about 6.0 μm ; and
 - d) a range of particle diameters of from about 0.78 μm to about 30 μm .

55. The method of Claim 54, comprising grinding the fly ash with a grinding medium in a non-expanded bed, wherein the volume of fly ash is less than the void volume of the grinding medium.
- 5 56. The method of Claim 55, wherein the ratio of fly ash to grinding medium is about 1 part fly ash to about 4 parts grinding medium, by volume.
57. The method of Claim 55, wherein the ratio of fly ash to grinding medium is about 1 part fly ash to about 18 parts grinding medium, by weight.
- 10 58. The method of Claim 55, wherein the grinding medium comprises stainless steel.
59. The method of Claim 55, wherein the grinding medium comprises carbon steel.
- 15 60. The method of Claim 54, wherein the fly ash is dry bottom boiler fly ash.
61. The method of Claim 54, wherein the fly ash is wet bottom boiler fly ash.

ABSTRACT OF THE DISCLOSURE

The present invention relates to concrete, mortar and other hardenable mixtures comprising cement and fly ash for use in construction. The invention relates to hardenable mixtures comprising cement and fly ash which can achieve greater compressive strength than hardenable mixtures containing only concrete over the time period relevant for construction. In a specific example, processing fly ash by grinding to the desired size distribution range gives a 100% yield of fly ash for utilization in high quality concrete and mortar. Such materials can thus significantly reduce construction costs, as well as provide a useful outlet for material that is generally regarded as a pollutant and a nuisance.

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Compressive Strength of Fly Ash Mortar (M8F9525)

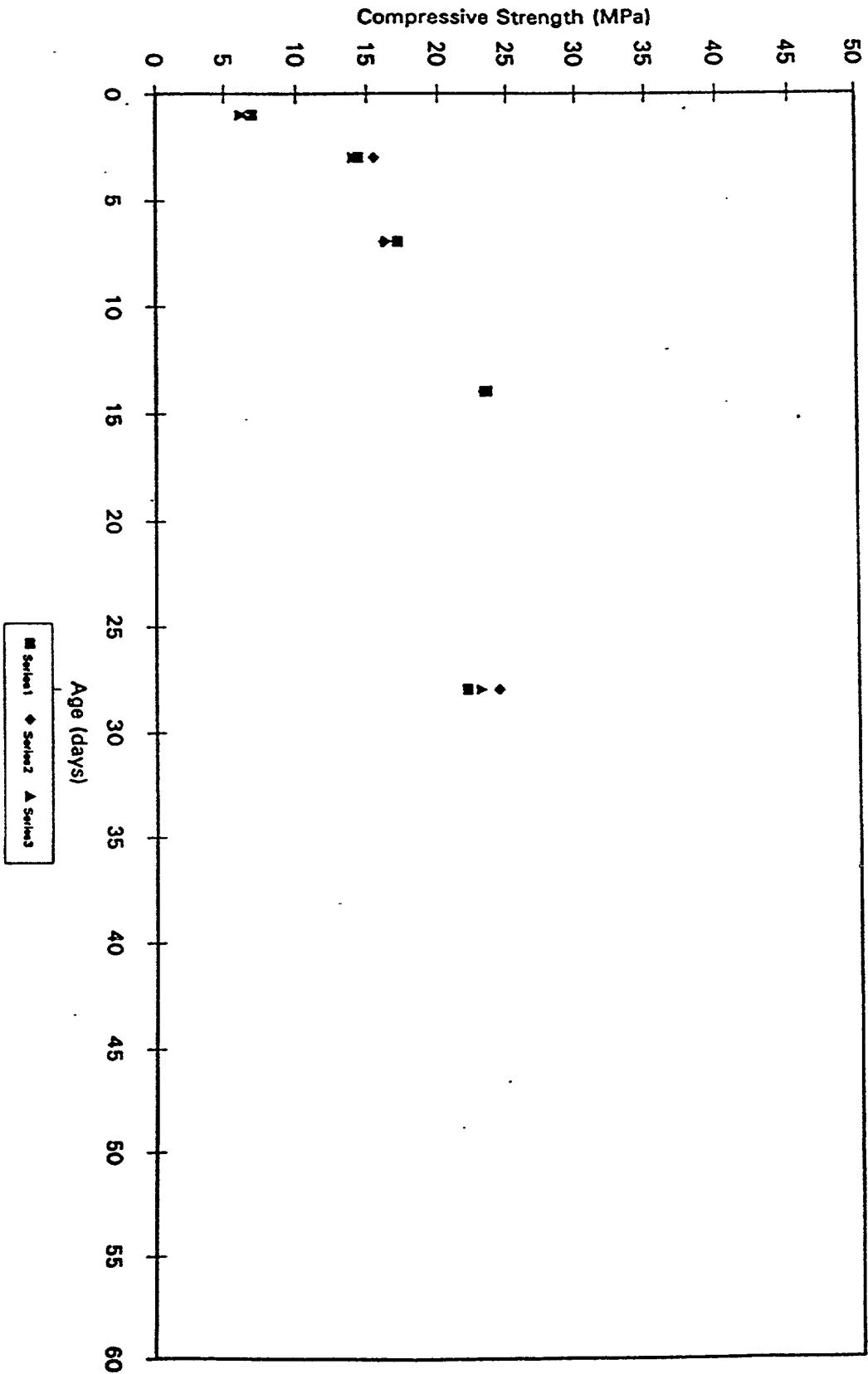


Figure 1

Compressive Strength of Fly Ash Mortar (M9F9525)

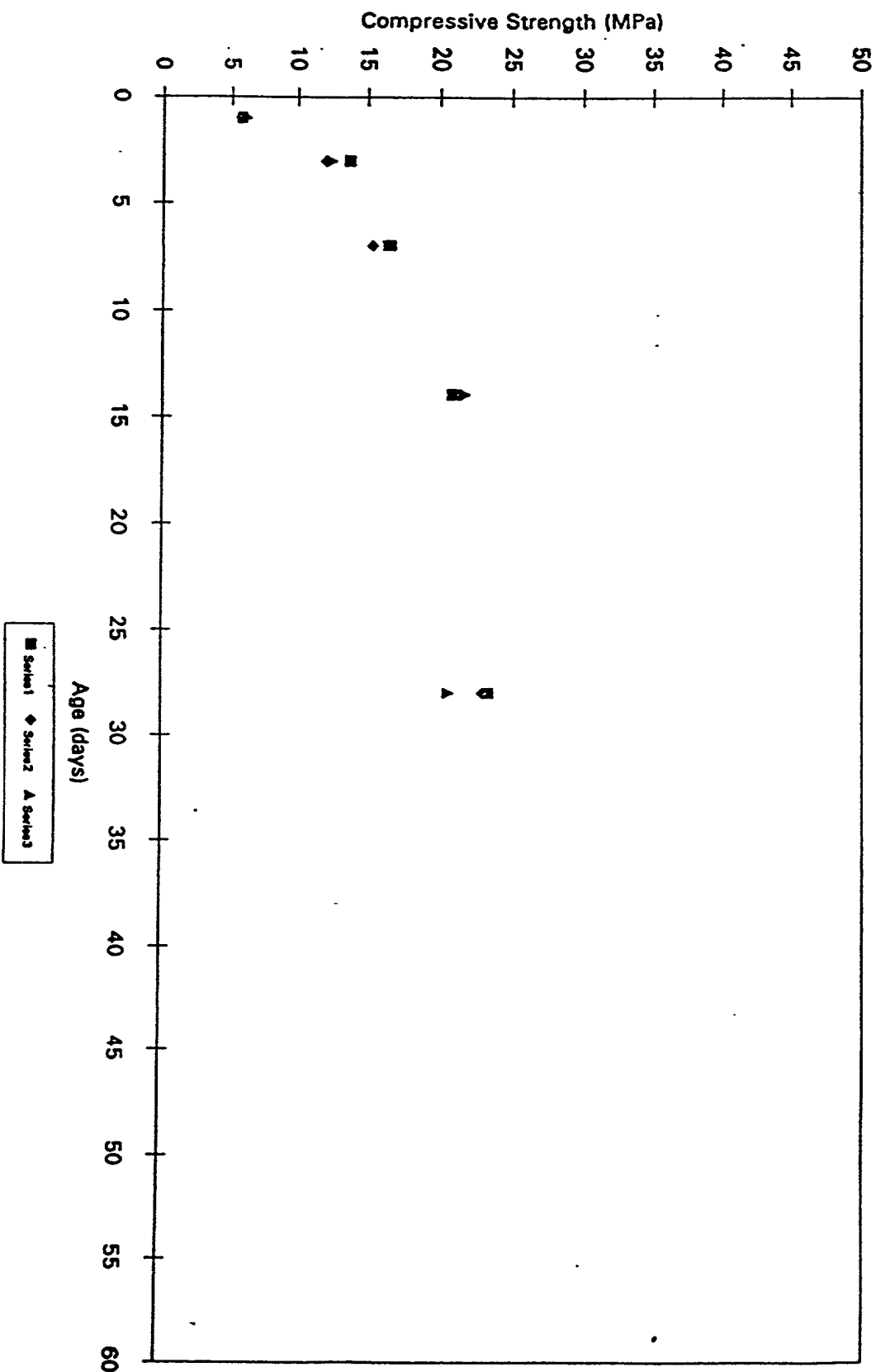


Figure 2

Compressive Strength of Fly Ash Mortar (M8F9535)

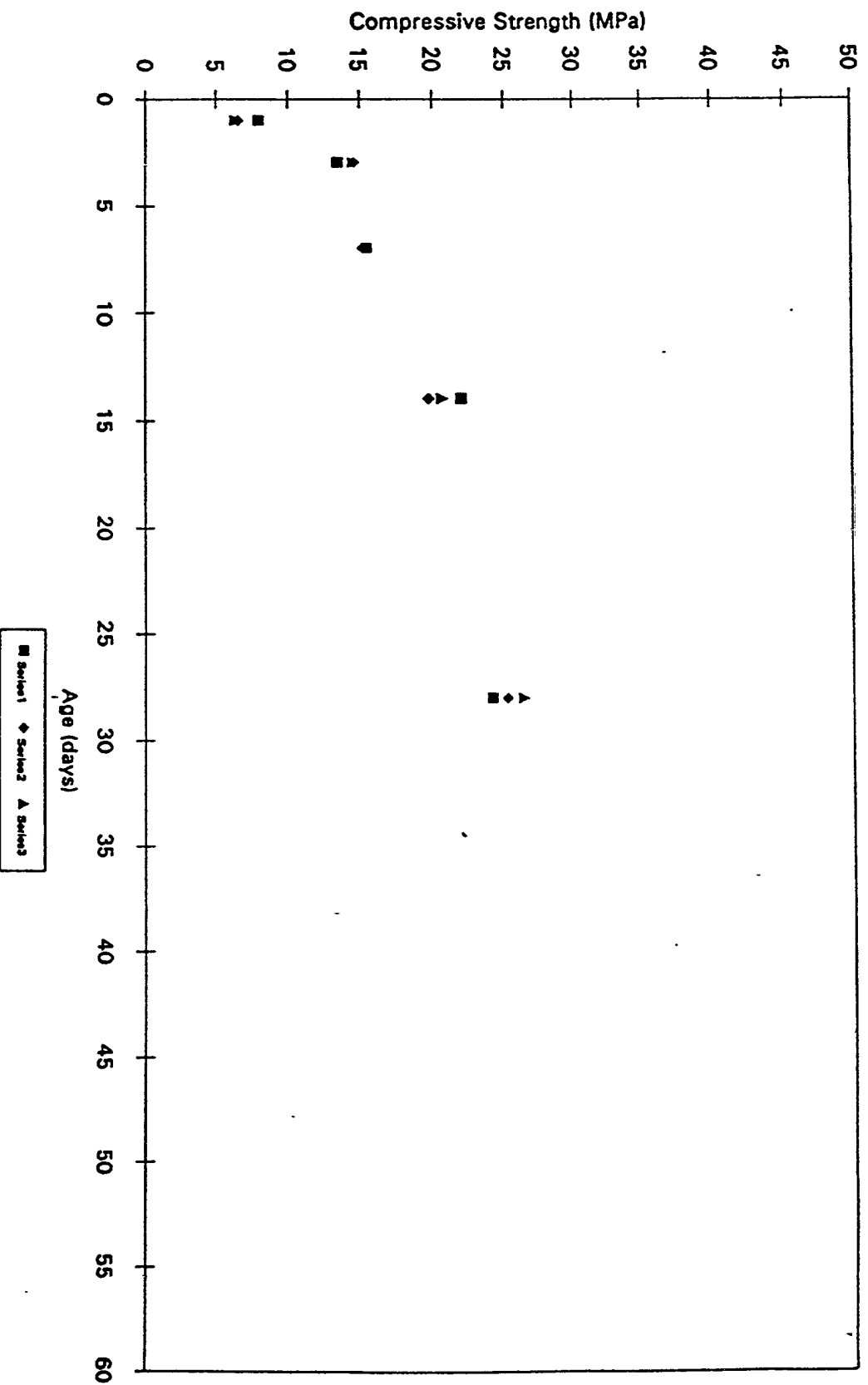


Figure 3

090344E10 012109

Compressive Strength of Fly Ash Mortar (M9F9535)

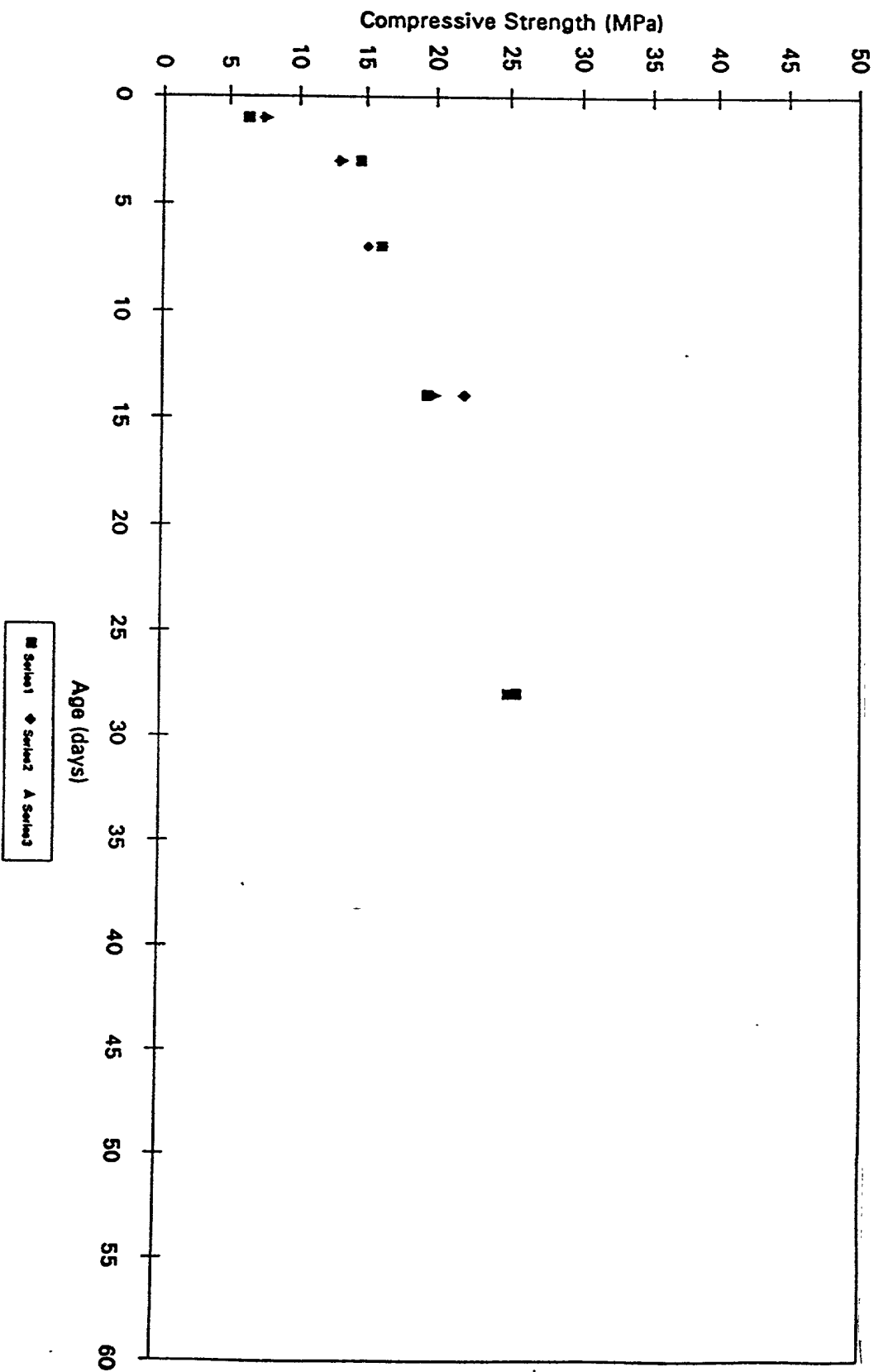


Figure 4

Compressive Strength of Fly Ash Mortar (M13N9425)

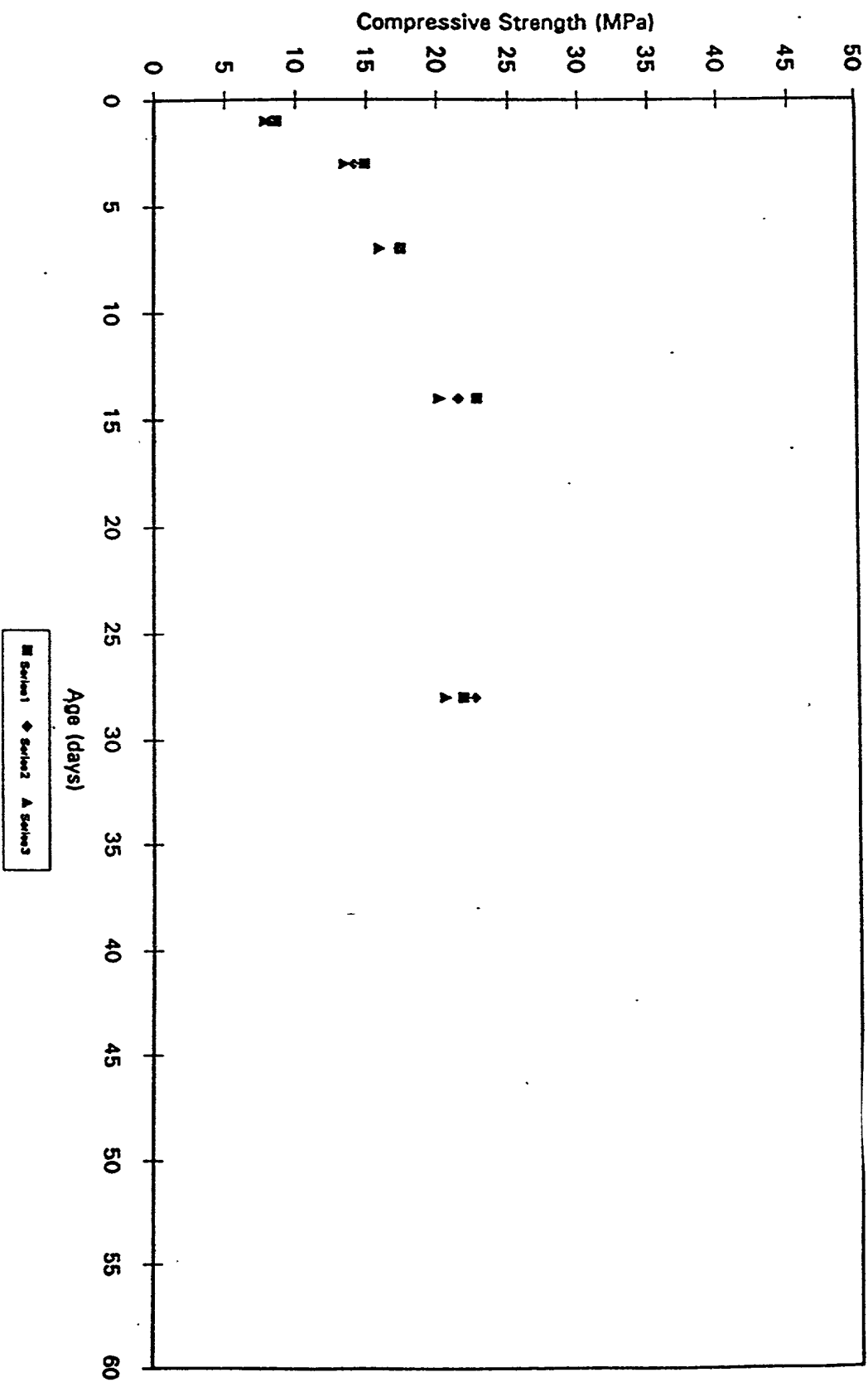


Figure 5

The Dry Grinding Process

The Attritor dry grinding process is achieved in a stationary tank by means of a rotating shaft and arms that agitate the media into a random state of motion of internal porosity called kinematic porosity. In this expanded condition, the media and particles are free to move, collide and impinge upon each other.

FIG. 6 A. KINEMATIC POROSITY

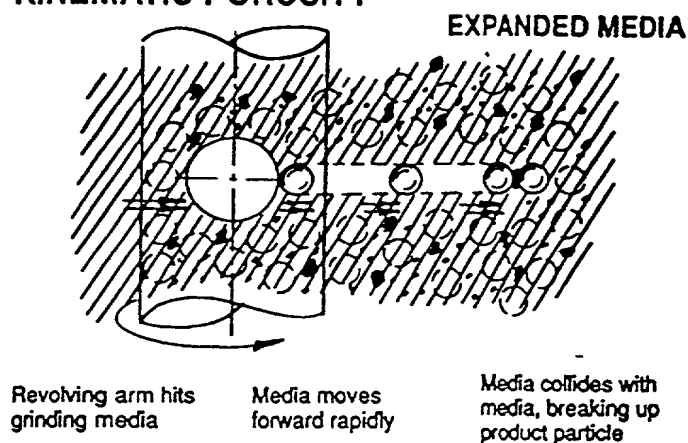


FIG. 6 B. MEDIA AT REST

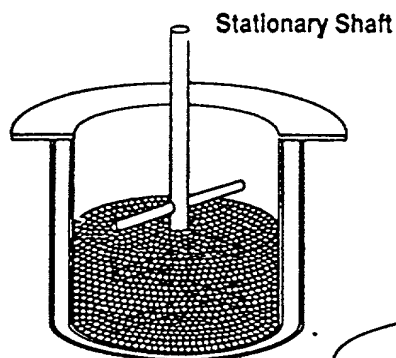


FIG. 6 C.

EXPANDED MEDIA
Rotating Shaft

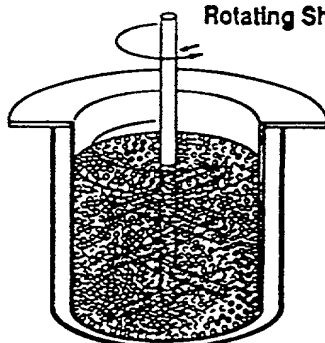


Figure 6

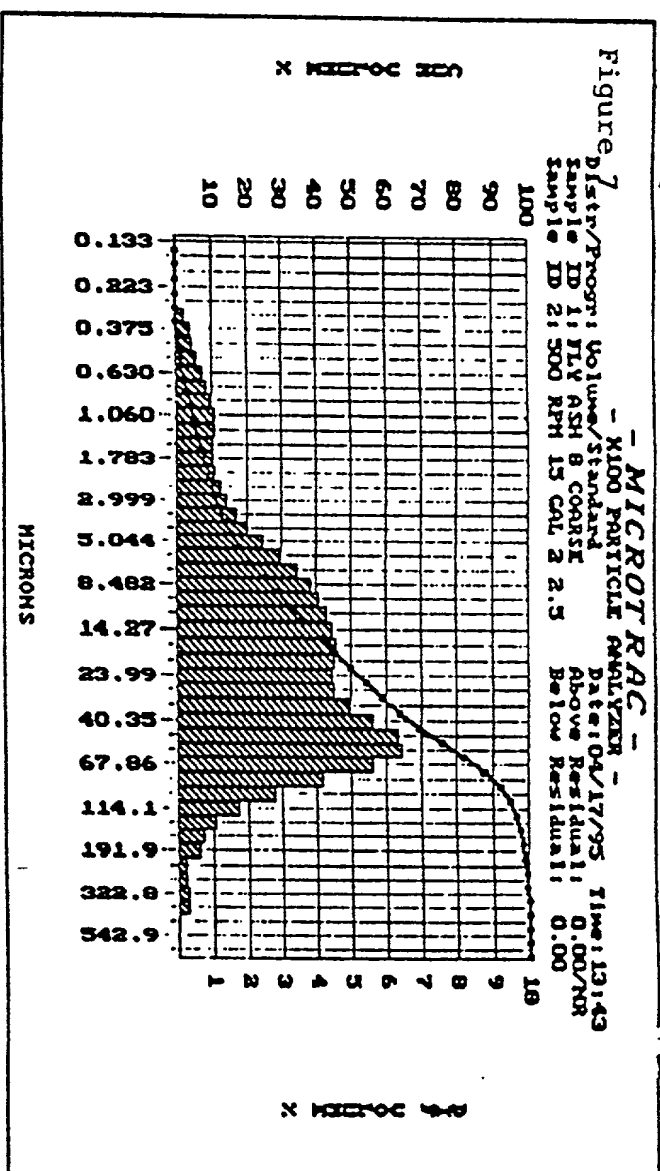


Figure 7

00334310 043430

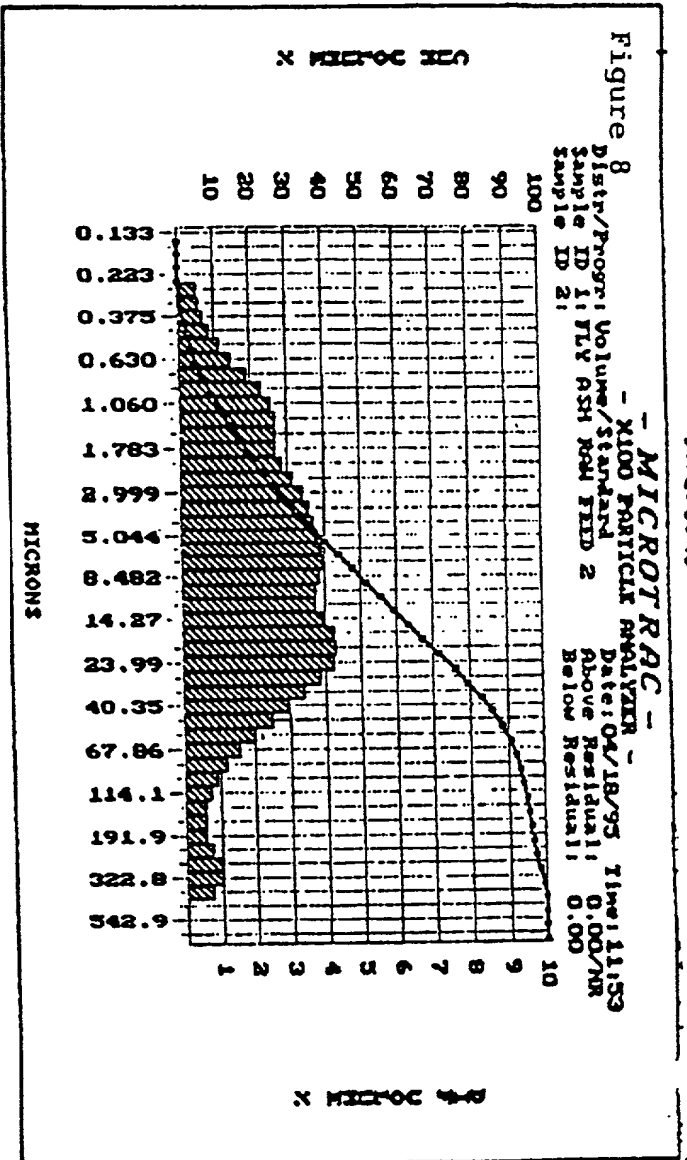


Figure 8

[illegible]

715-1-060 CIP (Sheet 9 of 23)
Compressive Strength of Fly Ash Mortar
(MOG9525)

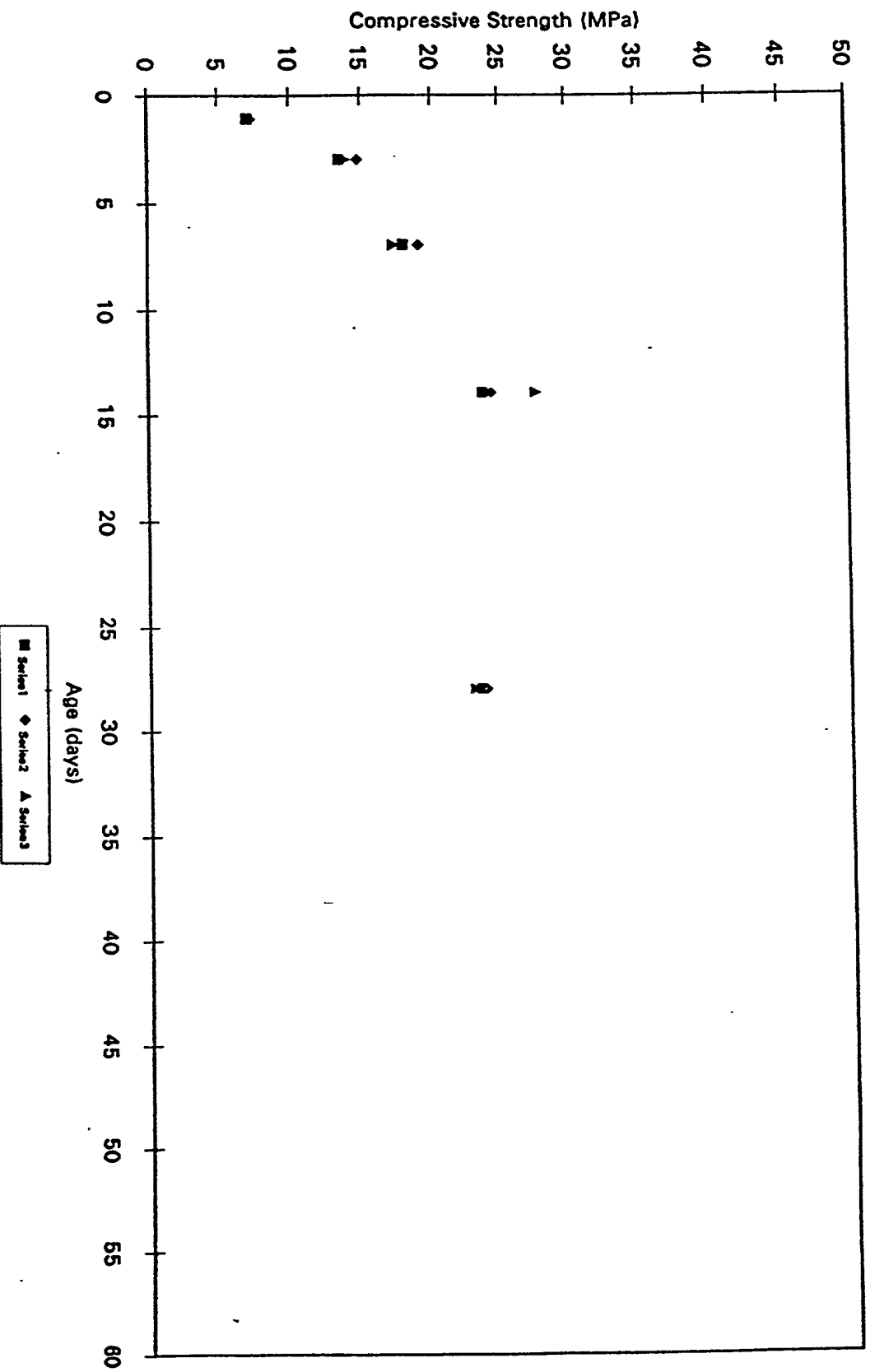


Figure 9

002234340 012169

Compressive Strength of Fly Ash Mortar (M8CG225)

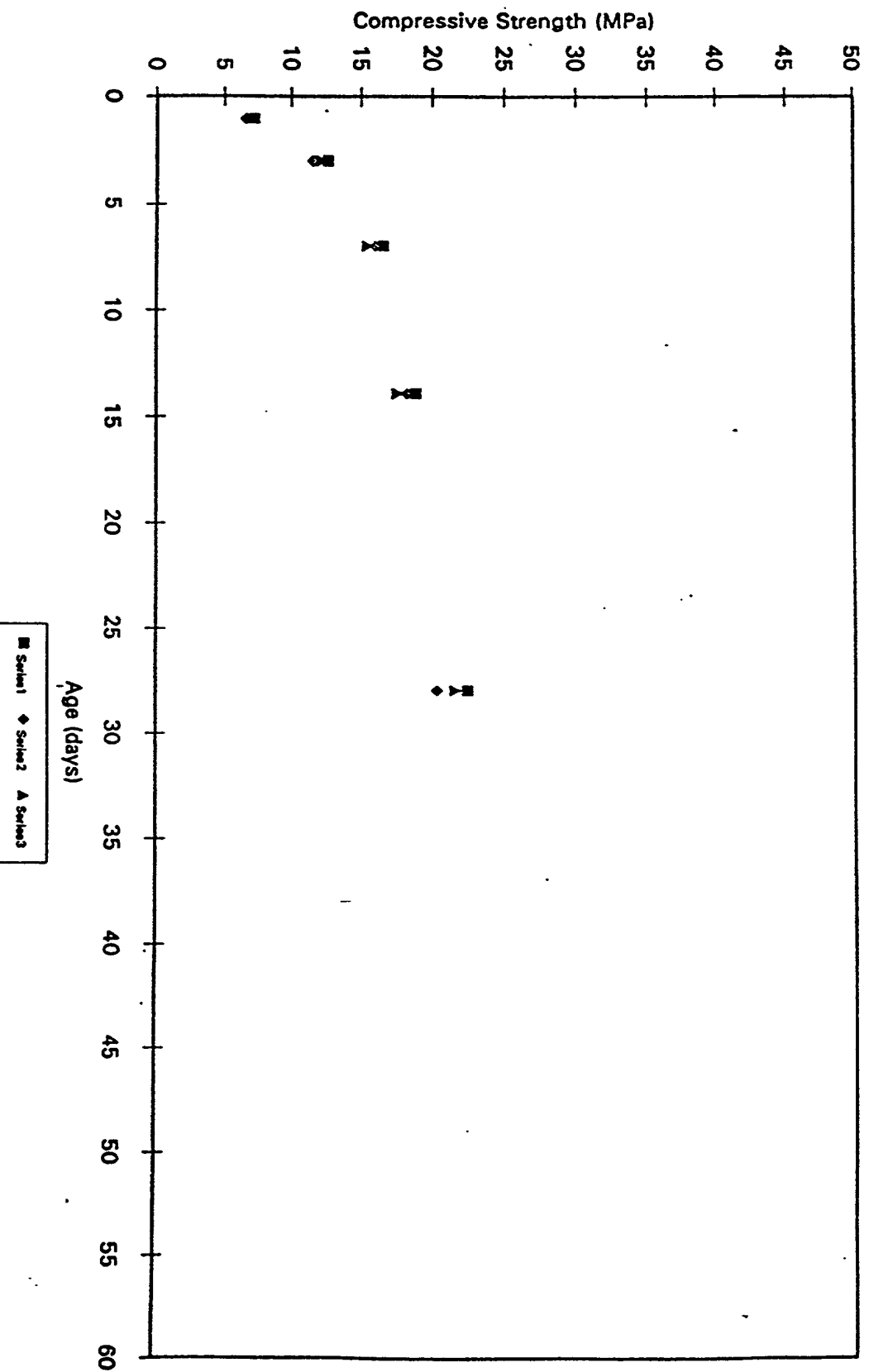


Figure 10

Compressive Strength of Fly Ash Mortar (MOG9535)

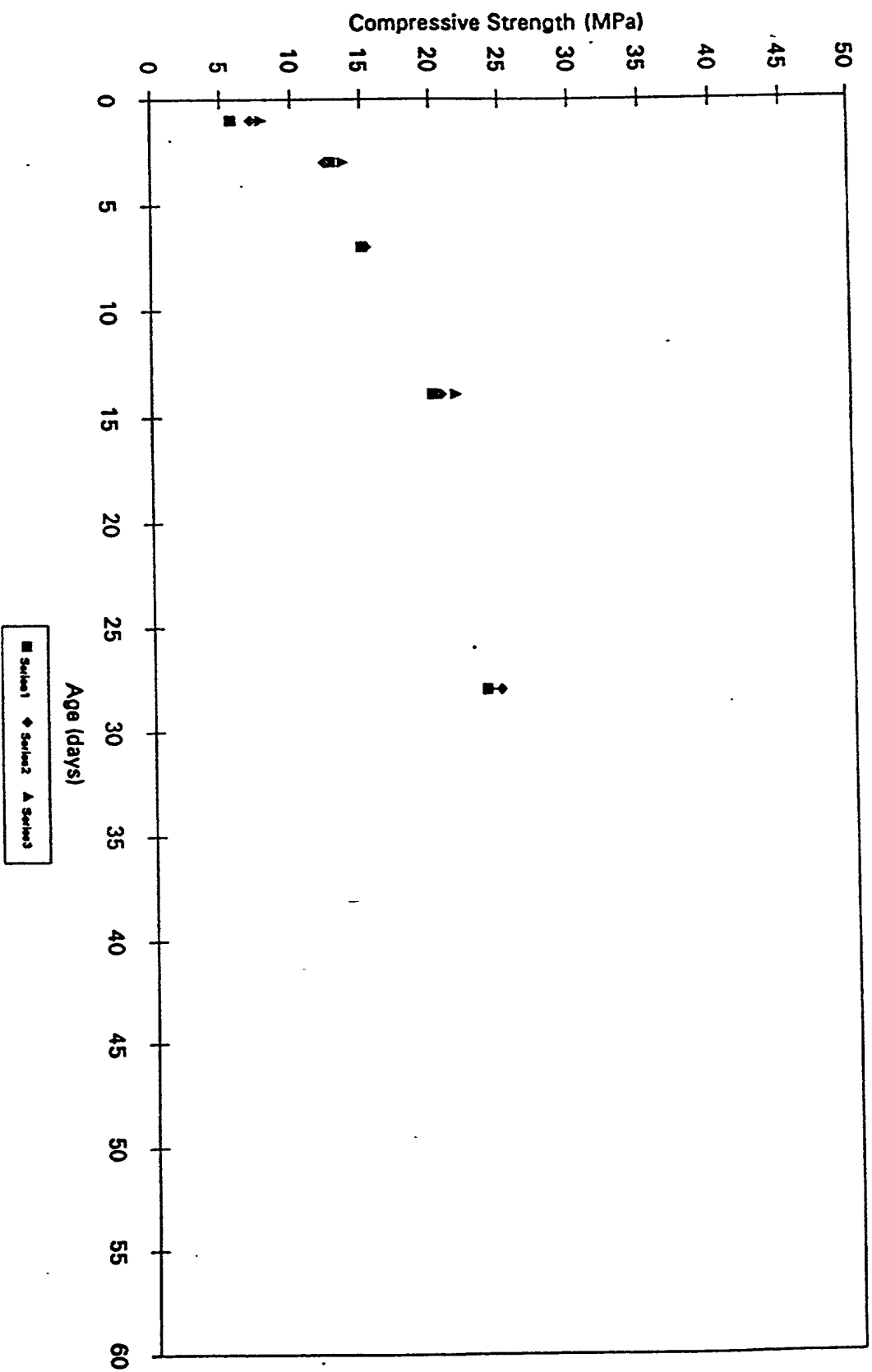


Figure 11

715-1-060 CIP (Sheet 12 of 23)
Compressive Strength of Control Mortar
(CC)

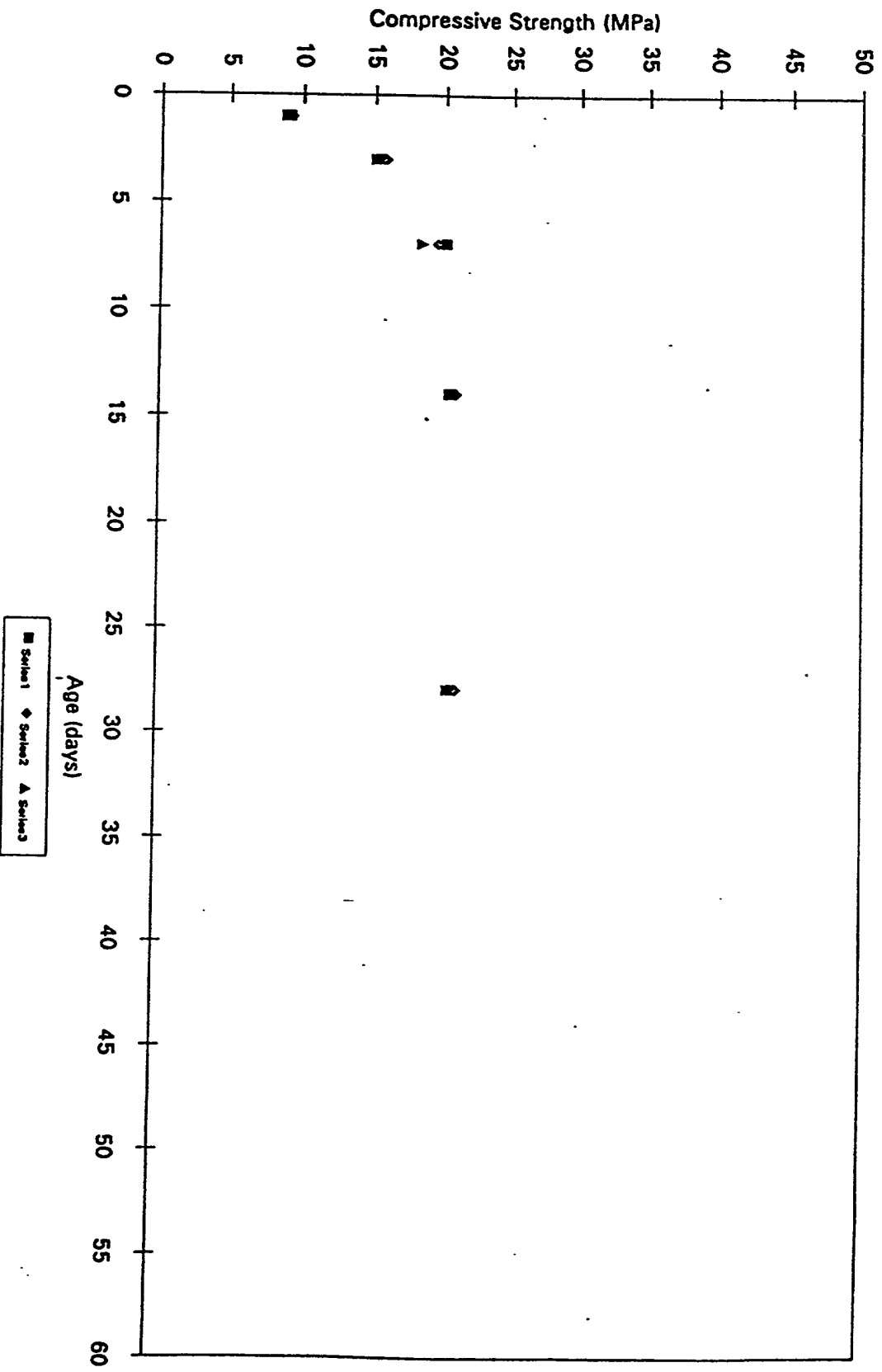


Figure 12

09:34:31 04/14/19

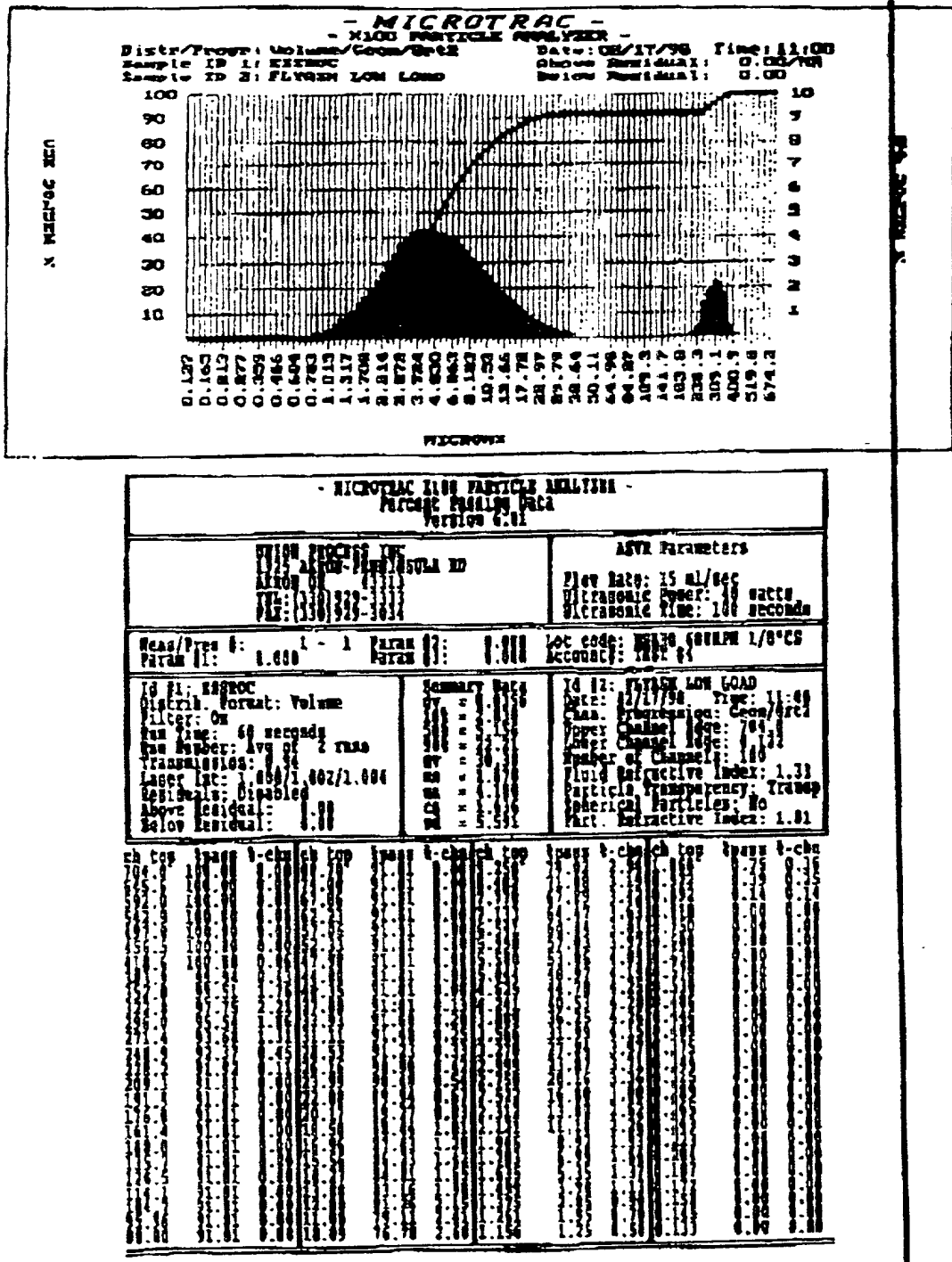
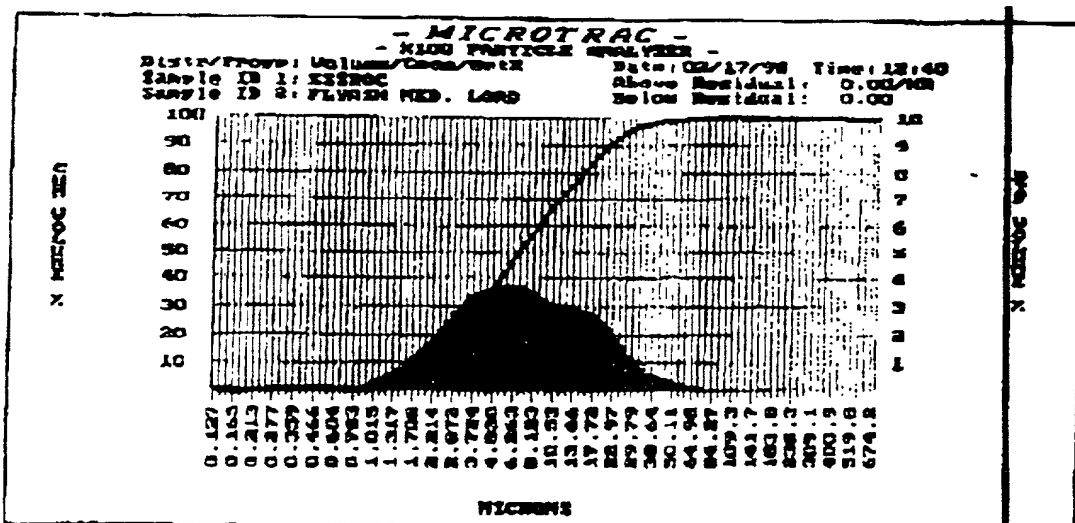


Figure 13



- MICROTRAC X100 PARTICLE ANALYZER - Percent Passing Data Version 6.01			
SYSTEM PARAMETERS 11111 ARROW-PLUS 11111 ARROW-PLUS 11111 ARROW-PLUS		ANAL Parameters Flow Rate: 15 ml/sec Ultrasonic Power: 1 watts Ultrasonic Time: 100 seconds	
Mean/Drop #: 1 - 1	Param #1: 0.000	Param #2: 0.000	Lot code: 00100 60000 1/0/CS
Param #1: 0.000	Param #3: 0.000	Account: 1000 05	
ID #1: KISROC Distrib. Param: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 1 runs Transmittance: 1.000/1.000 Laser Int: 0.000/1.000 Residual: Disabled Above Residual: 0.00 Below Residual: 0.00		Summary Data D1 = 0.000 D2 = 0.000 D3 = 0.000 D4 = 0.000 D5 = 0.000 D6 = 0.000 D7 = 0.000 D8 = 0.000 D9 = 0.000 D10 = 0.000 D11 = 0.000 D12 = 0.000 D13 = 0.000 D14 = 0.000 D15 = 0.000 D16 = 0.000 D17 = 0.000 D18 = 0.000 D19 = 0.000 D20 = 0.000 D21 = 0.000 D22 = 0.000 D23 = 0.000 D24 = 0.000 D25 = 0.000 D26 = 0.000 D27 = 0.000 D28 = 0.000 D29 = 0.000 D30 = 0.000 D31 = 0.000 D32 = 0.000 D33 = 0.000 D34 = 0.000 D35 = 0.000 D36 = 0.000 D37 = 0.000 D38 = 0.000 D39 = 0.000 D40 = 0.000 D41 = 0.000 D42 = 0.000 D43 = 0.000 D44 = 0.000 D45 = 0.000 D46 = 0.000 D47 = 0.000 D48 = 0.000 D49 = 0.000 D50 = 0.000 D51 = 0.000 D52 = 0.000 D53 = 0.000 D54 = 0.000 D55 = 0.000 D56 = 0.000 D57 = 0.000 D58 = 0.000 D59 = 0.000 D60 = 0.000 D61 = 0.000 D62 = 0.000 D63 = 0.000 D64 = 0.000 D65 = 0.000 D66 = 0.000 D67 = 0.000 D68 = 0.000 D69 = 0.000 D70 = 0.000 D71 = 0.000 D72 = 0.000 D73 = 0.000 D74 = 0.000 D75 = 0.000 D76 = 0.000 D77 = 0.000 D78 = 0.000 D79 = 0.000 D80 = 0.000 D81 = 0.000 D82 = 0.000 D83 = 0.000 D84 = 0.000 D85 = 0.000 D86 = 0.000 D87 = 0.000 D88 = 0.000 D89 = 0.000 D90 = 0.000 D91 = 0.000 D92 = 0.000 D93 = 0.000 D94 = 0.000 D95 = 0.000 D96 = 0.000 D97 = 0.000 D98 = 0.000 D99 = 0.000 D100 = 0.000	
ID #2: FLUOREN MED. LOAD Date: 02/17/98 Time: 12:40 Run Progression: 0.00/100 Upper Channel Edge: 784 Lower Channel Edge: 111 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp Spherical Particles: No Part. Refractive Index: 1.01			

Figure 14

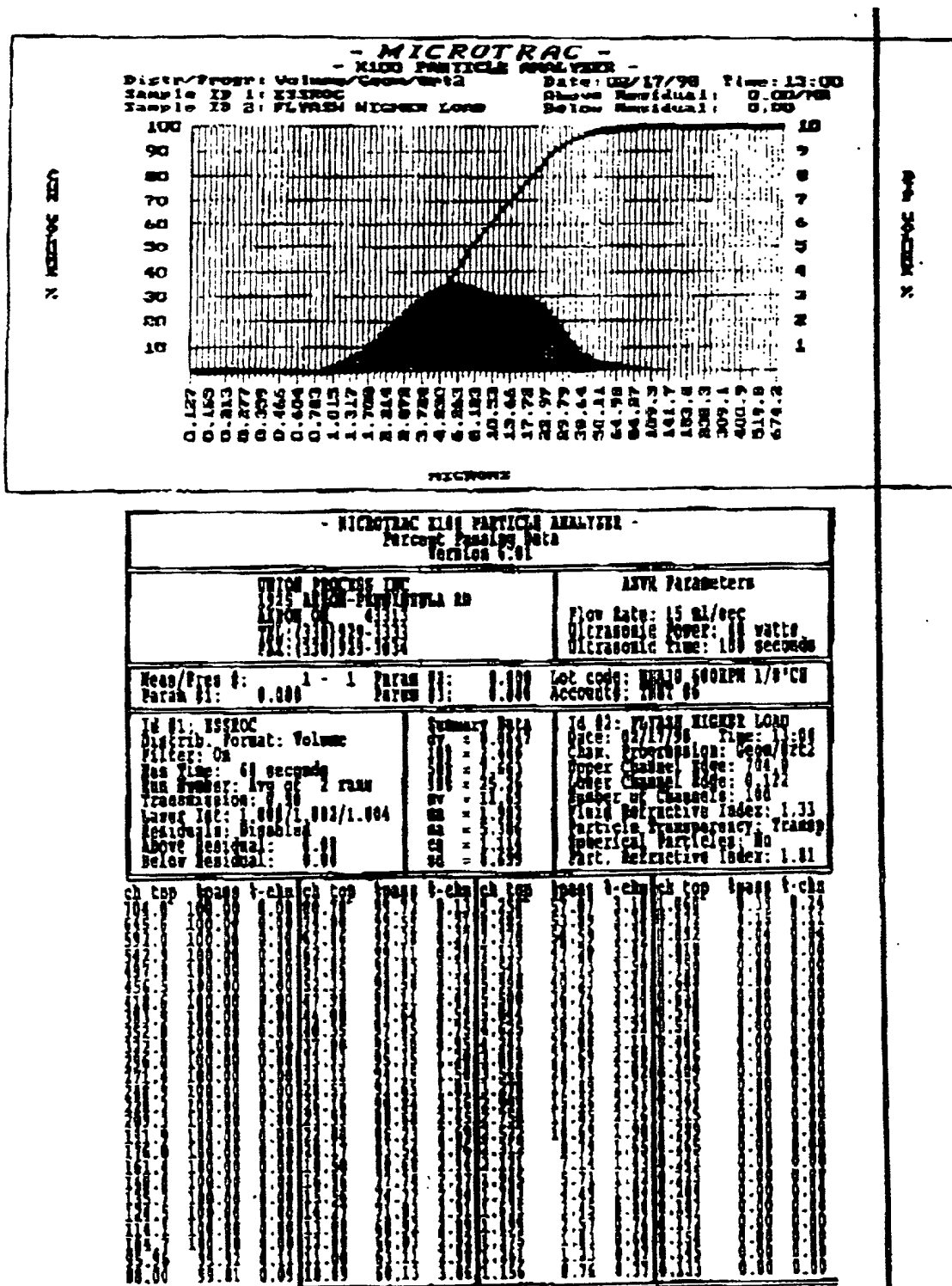


Figure 15

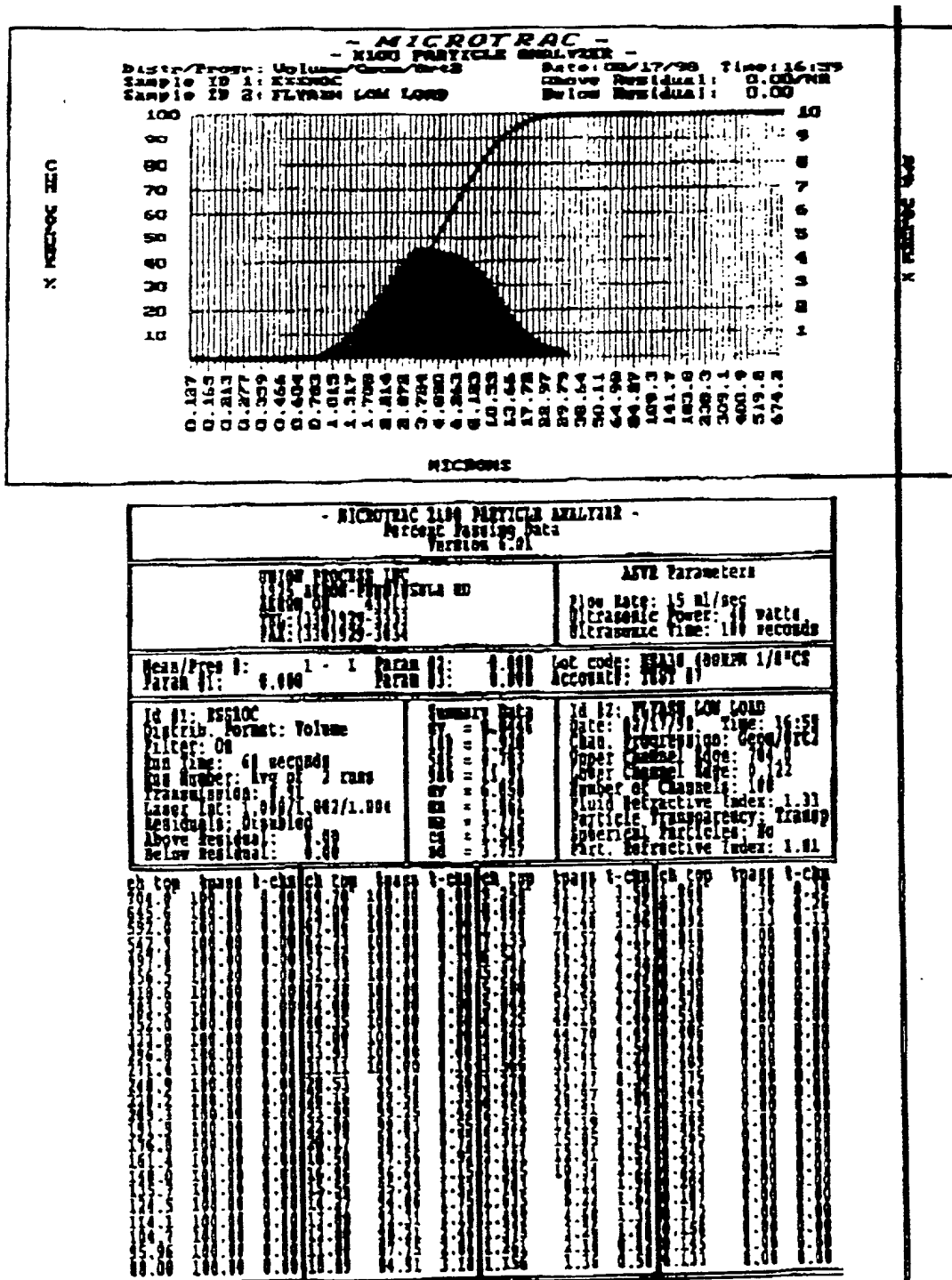


Figure 16

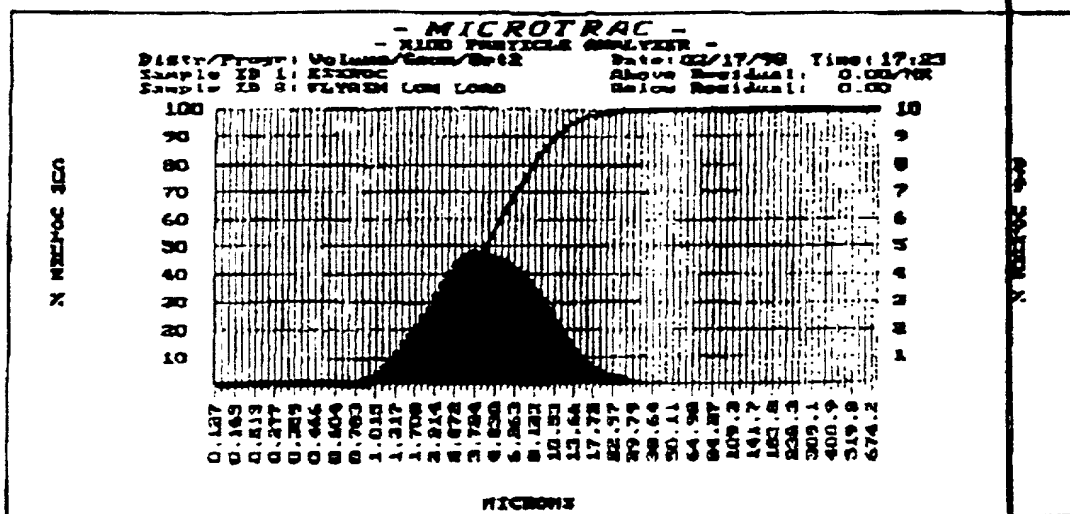
[illegible]

Figure 17

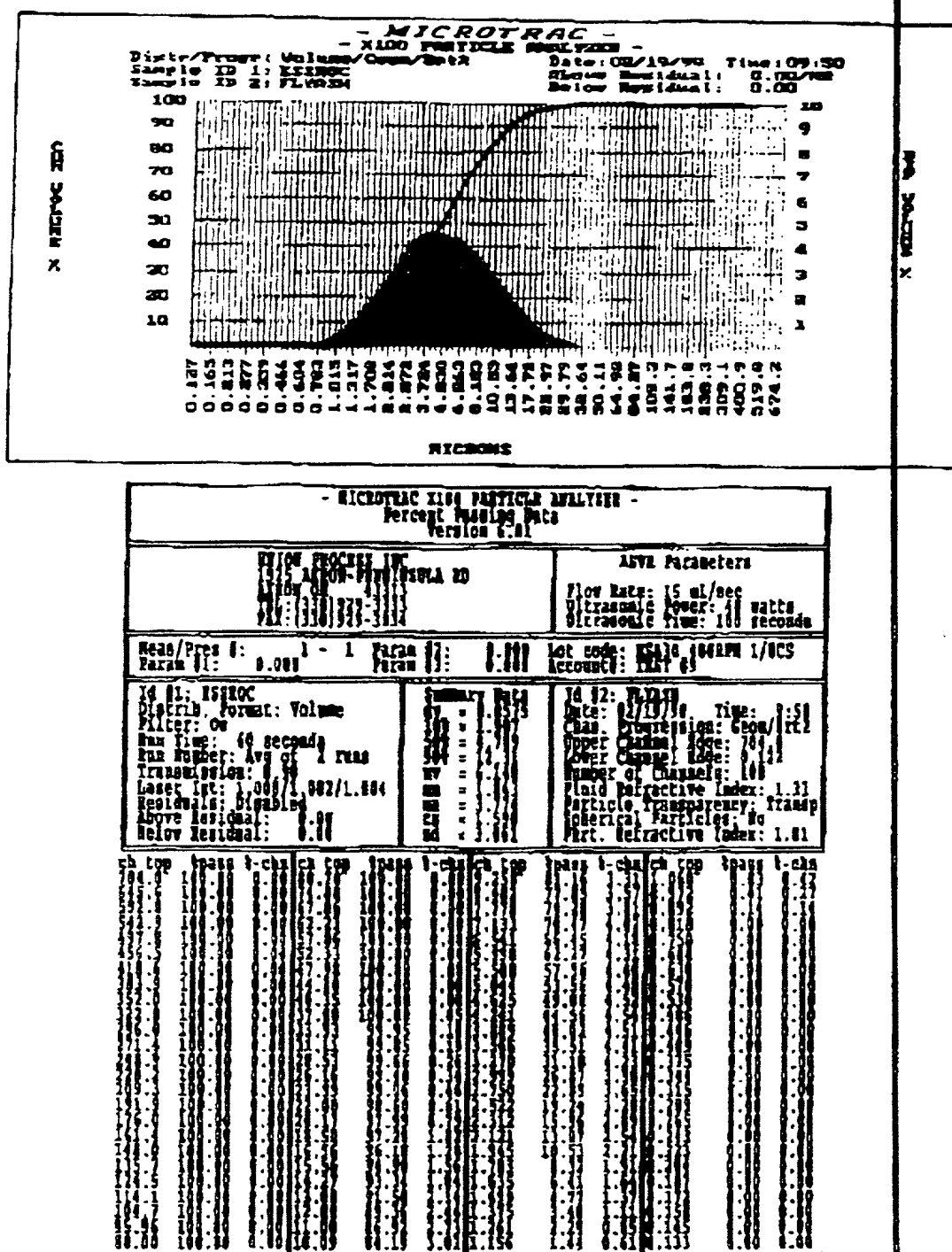


Figure 18

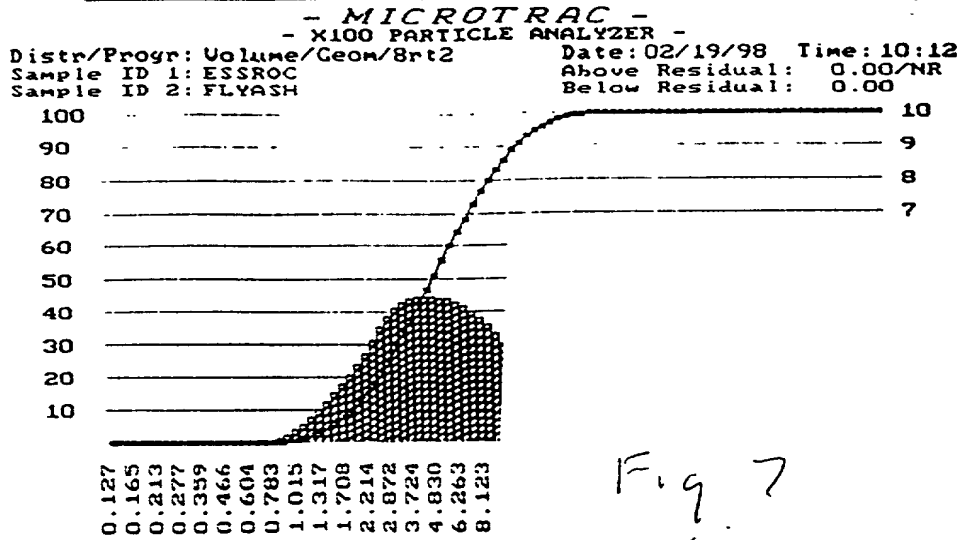


Fig 7
FLY

- MICROTRAC X100 F Percent Pass Versio											
UNION PROCESS INC 1925 AKRON-PENINSULA RD AKRON OH 43313 TEL: (330) 929-3333 FAX: (330) 929-3034						FLOW RATE: 40 watts Ultrasonic Power: 40 watts Ultrasonic Time: 100 seconds					
Meas/Pres #: 1 - 1			Param #2: 0.000			Lot code: HSA30 400RPM 1/8CS					
Param #1: 0.000			Param #3: 0.000			Account#: TEST #10					
Id #1: ESSROC Distrib. Format: Volume Filter: On Run Time: 60 seconds Run Number: Avg of 2 runs Transmission: 0.93 Laser Int: 1.003/1.002/1.004 Residuals: Disabled Above Residual: 0.00 Below Residual: 0.00				Summary Data dv = 0.0393 10% = 1.969 50% = 4.912 90% = 12.55 mv = 6.301 mn = 1.873 ma = 3.899 cs = 1.539 sd = 3.965				Id #2: FLYASH Date: 02/19/98 Time: 10:12 Chan. Progression: Geom/8rt2 Upper Channel Edge: 704.0 Lower Channel Edge: 0.122 Number of Channels: 100 Fluid Refractive Index: 1.33 Particle Transparency: Transp Spherical Particles: No Part. Refractive Index: 1.81			
ch top	tpass	%-chn	ch top	tpass	%-chn	ch top	tpass	%-chn	ch top	tpass	%-chn
704.0	100.00	0.00	80.70	100.00	0.00	9.250	79.94	3.55	1.060	0.75	0.37
645.6	100.00	0.00	74.00	100.00	0.00	8.482	76.39	3.79	0.972	0.38	0.25
592.0	100.00	0.00	67.86	100.00	0.00	7.778	72.60	4.00	0.892	0.13	0.13
542.9	100.00	0.00	62.23	100.00	0.00	7.133	68.60	4.17	0.818	0.00	0.00
497.8	100.00	0.00	57.06	100.00	0.00	6.541	64.43	4.28	0.750	0.00	0.00
456.5	100.00	0.00	52.33	100.00	0.00	5.998	60.15	4.37	0.688	0.00	0.00
418.6	100.00	0.00	47.98	100.00	0.00	5.500	55.78	4.41	0.630	0.00	0.00
383.9	100.00	0.00	44.00	100.00	0.00	5.044	51.37	4.45	0.578	0.00	0.00
352.0	100.00	0.00	40.35	100.00	0.00	4.625	46.92	4.46	0.530	0.00	0.00
322.8	100.00	0.00	37.00	100.00	0.00	4.241	42.46	4.44	0.486	0.00	0.00
296.0	100.00	0.00	33.93	100.00	0.00	3.889	38.02	4.39	0.446	0.00	0.00
271.4	100.00	0.00	31.11	100.00	0.17	3.566	33.63	4.28	0.409	0.00	0.00
248.9	100.00	0.00	28.53	99.83	0.28	3.270	29.35	4.09	0.375	0.00	0.00
228.2	100.00	0.00	26.16	99.55	0.35	2.999	25.26	3.83	0.344	0.00	0.00
209.3	100.00	0.00	23.99	99.20	0.48	2.750	21.43	3.49	0.315	0.00	0.00
191.9	100.00	0.00	22.00	98.72	0.65	2.522	17.94	3.11	0.289	0.00	0.00
176.0	100.00	0.00	20.17	98.07	0.86	2.312	14.82	2.75	0.265	0.00	0.00
161.4	100.00	0.00	18.50	97.21	1.11	2.121	12.07	2.39	0.243	0.00	0.00
148.0	100.00	0.00	16.96	96.10	1.37	1.945	9.68	2.07	0.223	0.00	0.00
135.7	100.00	0.00	15.56	94.73	1.67	1.783	7.61	1.78	0.204	0.00	0.00
124.5	100.00	0.00	14.27	93.04	1.98	1.635	5.83	1.51	0.187	0.00	0.00
114.1	100.00	0.00	13.08	91.08	2.30	1.499	4.32	1.25	0.172	0.00	0.00
104.7	100.00	0.00	12.00	88.78	2.63	1.375	3.07	1.00	0.158	0.00	0.00
95.96	100.00	0.00	11.00	86.15	2.95	1.261	2.07	0.77	0.145	0.00	0.00
88.00	100.00	0.00	10.09	83.20	3.26	1.156	1.30	0.55	0.133	0.00	0.00

Figure 19

601210 01812260

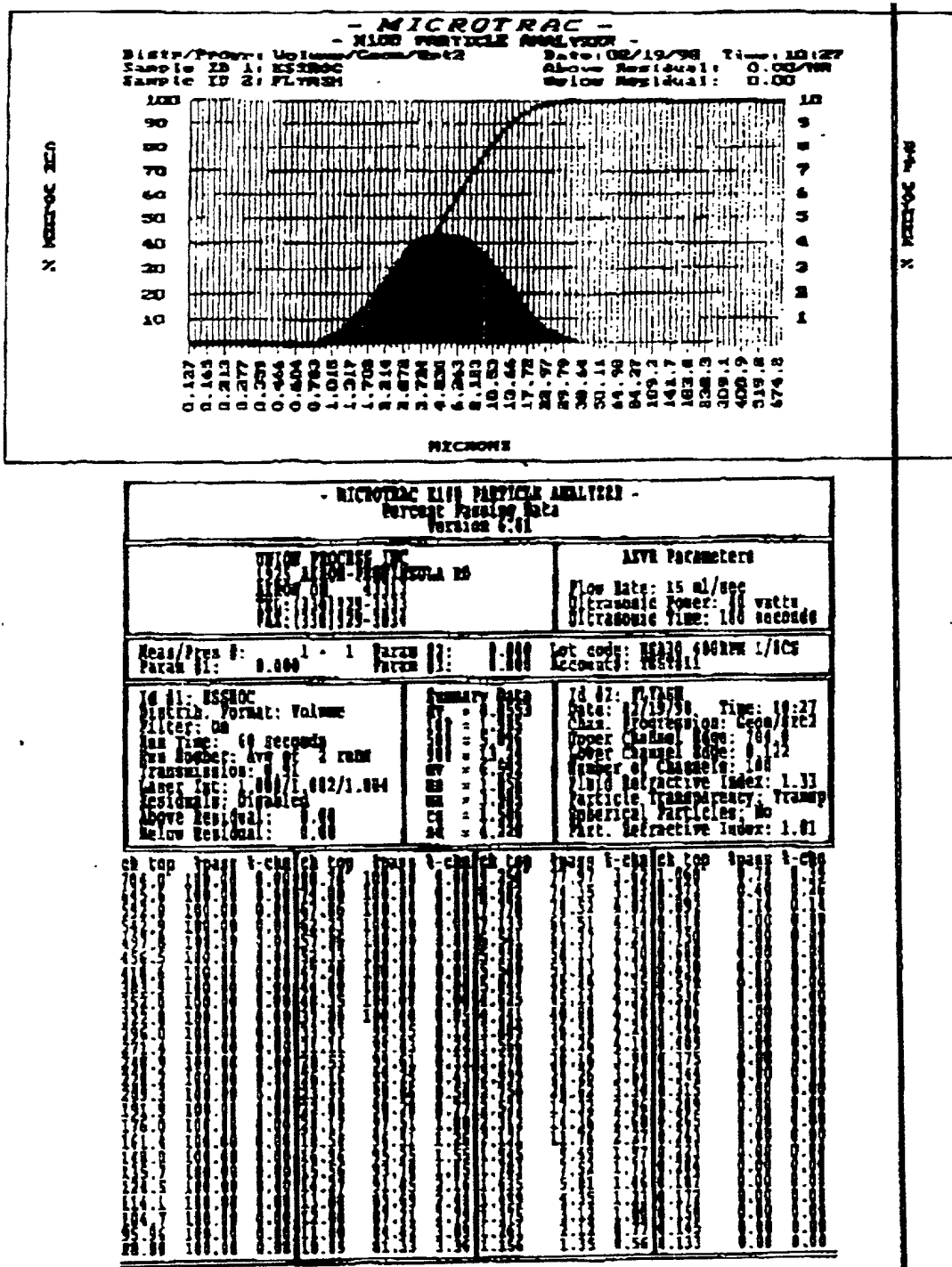


Figure 20

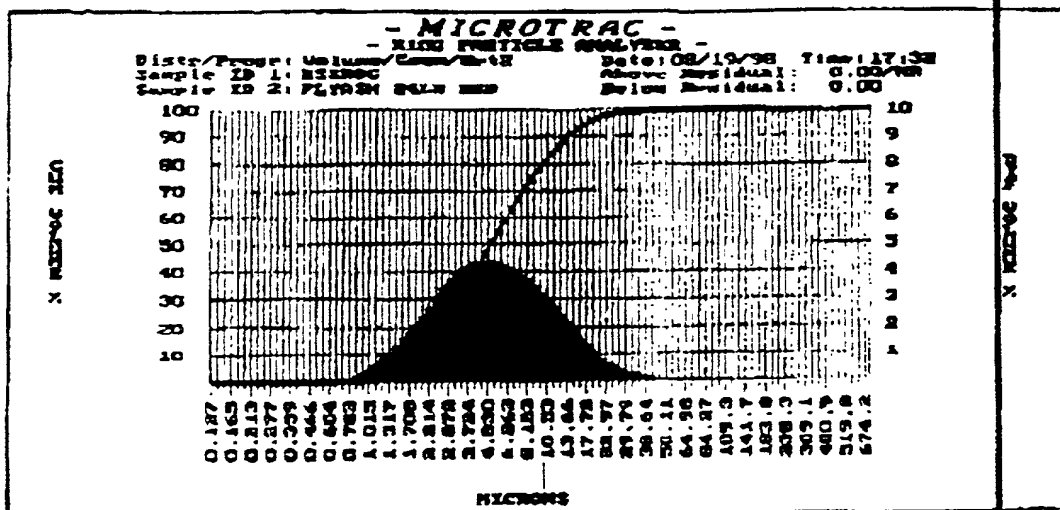
[illegible]

Figure 21

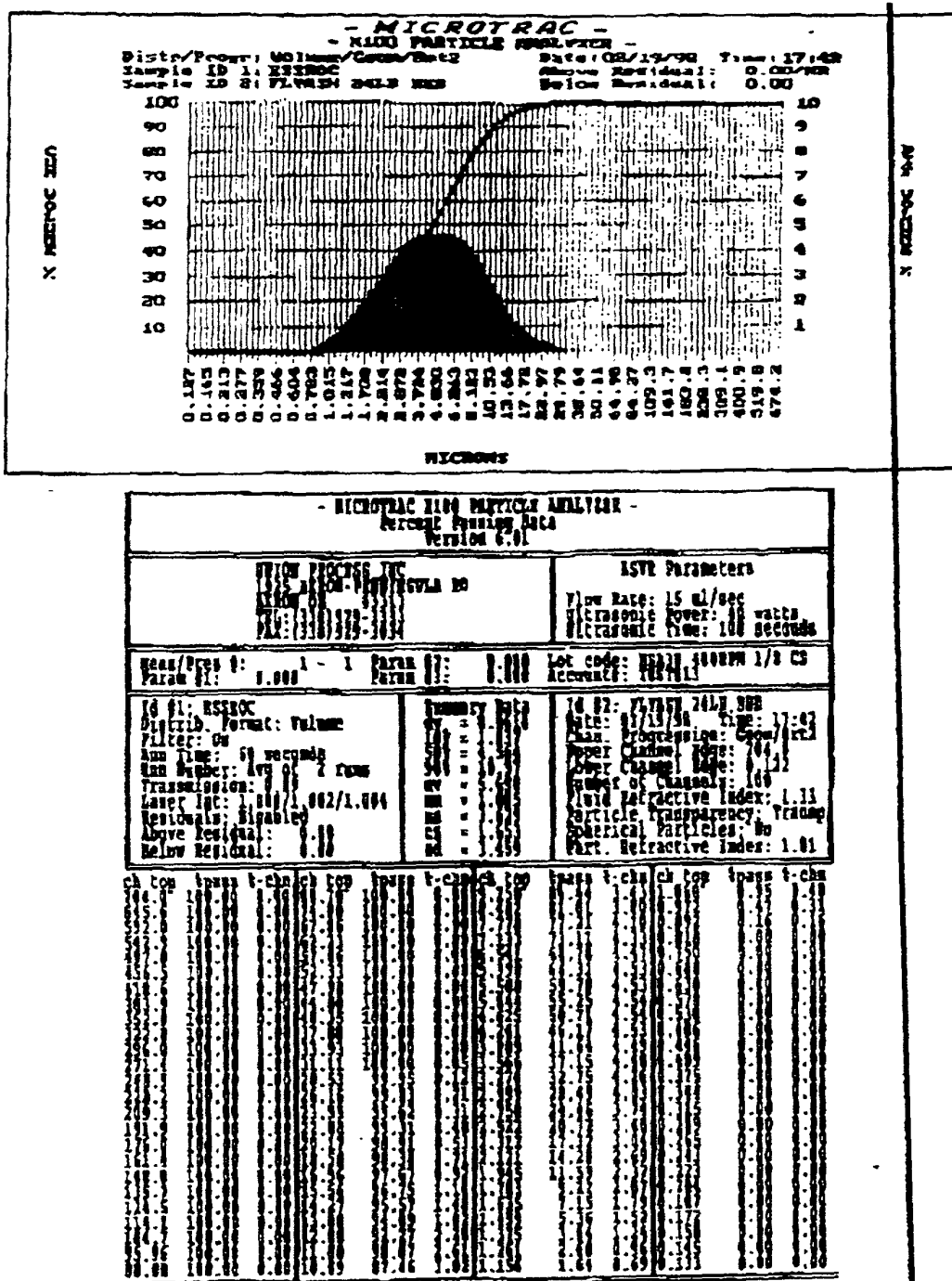


Figure 22

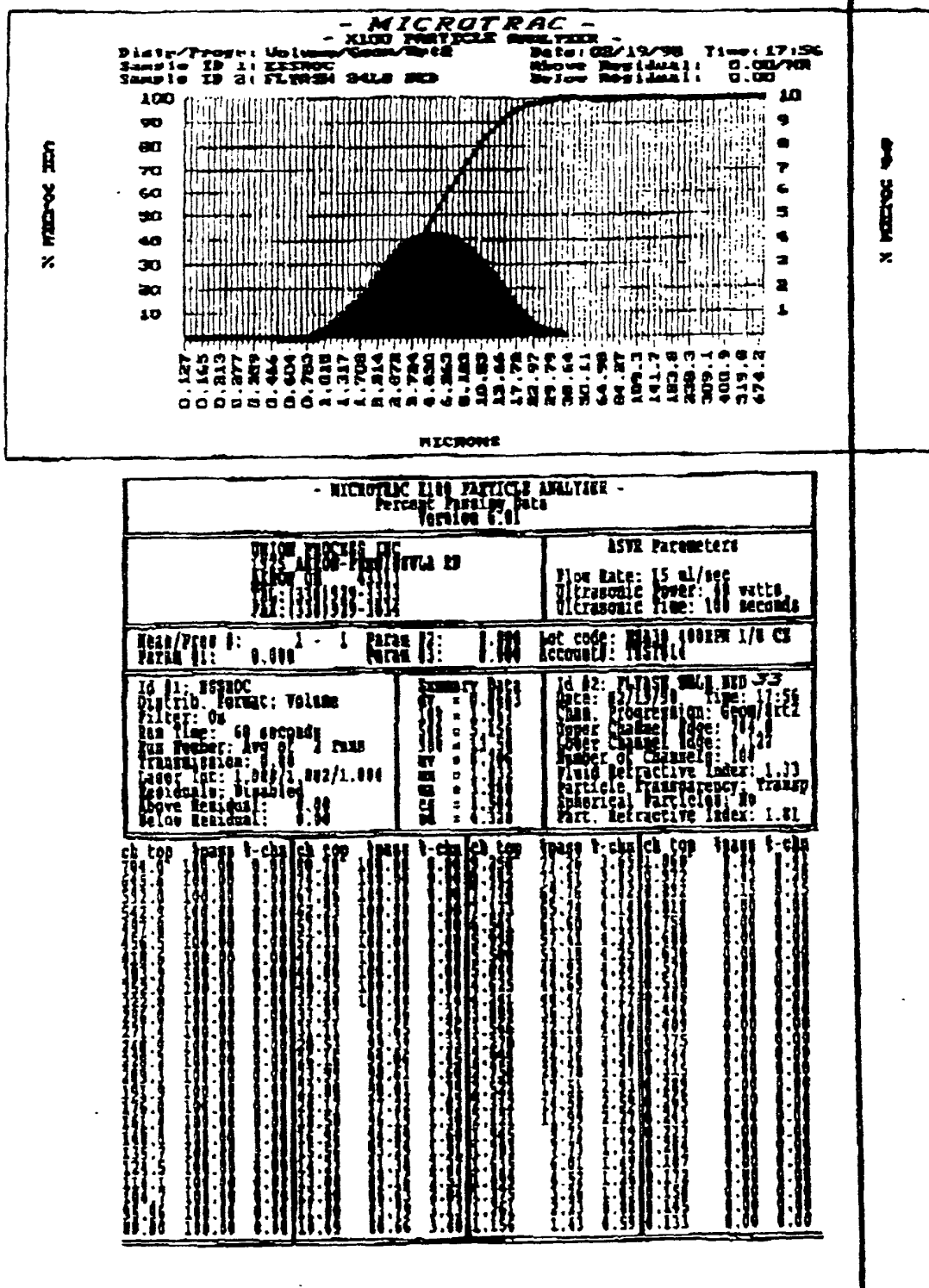


Figure 23

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

As below named inventors, we hereby declare that:

Our residence, post office address and citizenship are as stated below under our names.

We believe that we are the original, first and joint inventors of the subject matter which is claimed and for which a patent is sought on the invention entitled

METHOD FOR PREPARING FLY ASH FOR HIGH COMPRESSIVE STRENGTH CONCRETE AND MORTAR, AND COMPOSITIONS THEREOF

the Specification of which

☒ is attached hereto
☐ was filed on _____
as Application Serial No. _____
and was amended on _____ (if applicable).

We hereby state that we have reviewed and understand the contents of the above-identified Specification, including the claims, as amended by any amendment referred to above.

We acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, 1.56(a).

We hereby claim foreign priority benefits under Title 35, United States Code, §119 of any provisional application filed in the United States in accordance with 35 U.S.C. §1.119(e), or any application for patent that has been converted to a Provisional Application within one (1) year of its filing date, or any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

<u>APPLICATION</u> <u>NUMBER</u>	<u>COUNTRY</u>	<u>PRIOR FILED APPLICATION(S)</u> <u>(DAY/MONTH/YEAR FILED)</u>	<u>PRIORITY</u> <u>CLAIMED</u>
60/008,690	USA	12 DECEMBER 1995	YES

We hereby claim the benefit under Title 35, United States Code, §120 of any United States application listed below, and, insofar as the subject matter of each of the claims of this application is not disclosed in any prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a), which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION NO. _____	FILING DATE (DAY/MONTH/YEAR) _____	STATUS - PATENTED, PENDING, ABANDONED _____
08/768,167	13 DECEMBER 1996	PENDING

We hereby appoint as our attorneys or agents the following persons: Jack Matalon, (Attorney, Registration No. 22,441); Stefan J. Klauber (Attorney, Registration No. 22,604); David A. Jackson (Attorney, Registration No. 26,742); Michael D. Davis (Attorney, Registration No. 39,161); Joseph M. Homa (Attorney, Registration No. 40,023); Christine E. Dietzel (Agent, Registration No. 37,309); William C. Coppola (Attorney, Registration No. 41,686); Mark S. Cohen (Attorney, Registration No. 42,425); Donald J. Cox, Jr. (Attorney, Registration No. 37,804); Steven B. Stein (Attorney, Registration No. 43,159); Yuan Kong (Agent, Registration No. P-43,728); and Michael A. Yamin (Agent, Registration No. P-44,414), said attorneys or agents with full power of substitution and revocation to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

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We hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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SIGNATURE OF INVENTOR _____

DATE _____

08/768,167

Attorney Docket No: 715-1-060 CIP

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DATE _____